Beet-Sugar Handbook

Mosen Asadi, PhD



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Beet-Sugar Handbook



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Mosen Asadi, PhD



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DEDICATION

This book is dedicated to the following five sugar-technology masters, who contributed to establish the foundation of modern beet-sugar technology:

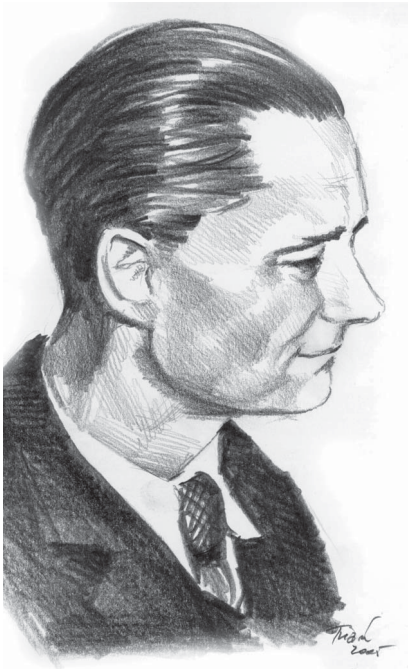
Richard A. McGinnis (1903–1995, from the United States) was the editor of *Beet-Sugar Technology*, a well-known sugar reference book, and the author of "Picking Table," a sugar-related column, in the *Sugar Journal* for 17 years. Dr. McGinnis most lasting achievement was the cofounding of the Beet-Sugar Institute in Fort Collins, Colorado, in 1972 with his friend James H. Fischer. The Institute teaches beet-sugar technology in two sections: beet-end and sugar-end. Later, this institute was renamed the **McGinnis Beet-Sugar Institute** to honor him.



Pavel M. Silin (1887–1967, from Russia) taught sugar technology at the Moscow Institute of Food Technology. Professor Silin was the author of *Technology of Beet-Sugar Production and Refining* and an outstanding researcher on many areas in beet-sugar technology, such as the diffusion process and molasses exhaustibility. Most sugar technologists know him through the Silin number (the length in meters of 100 grams of cossettes), which is used in sugar factories to justify the quality of beet cossettes. Professor Silin's daughter, Z. A. Silina, continued in her father's footsteps at the Moscow Institute of Food Technology and conducted research on the formation and exhaustion of molasses.



Jaroslav Dedek (1890–1962, born in Prague, Czech Republic, and died in Belgium) is the author of a book on juice carbonation (La Carbonate de Chaux) and several sugar-technology articles. A master of lime-carbondioxide chemistry, he could be called the father of the juice-purification process, the heart of the beet-sugar factory. His most important accomplishment, together with his colleague Josef Vašatko, at the Czech Institute in Brno, was the progressive preliming process.



Ferdinand Schneider (1911–1984, from Germany) is the author of the German edition of *Sugar Technology* and hundreds of sugar-related articles. Professor Schneider was director of Agricultural Technology and the Sugar Industry in Braunschweig, Germany, where he taught sugar technology for many years. During his career, he trained many men and women from around the world who have PhD in sugar technology.



Rudolf Bretschneider (1914–1985, from Czech Republic) is the author of the Czech edition of *Sugar Technology* and a few other books. Professor Bretschneider taught sugar technology in the Department of Carbohydrates Chemistry and Technology at the Institute of Chemical Technology (ICT), Prague, Czech Republic.

The author of this book is honored to have been Professor Bretschneider's graduate student from 1976 to 1984 in the Department of Carbohydrates Chemistry and Technology at ICT.



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PREFACE

Life starts when we begin to help others

This book is a unique learning reference to cover the void existing in beet-sugar literature. For the first time, the following subjects are covered in a single book:

- Basics of beet-sugar technology
- Sugarbeet farming
- Sugarbeet processing
- Laboratory methods of analysis
- Sugar-related tables used in calculations
- Basics of science related to sugar technology

The book has 12 chapters, an apendix, 18 sugar-related tables, and a complete glossary of technical terms. To provide an easier understanding of the subjects, it is written:

- **Effectively:** Subjects are given practically with less emphasis on reference citation
- **User-friendly:** Subjects are given systematically to ease readers' understanding
- **Conversationally:** Concepts are given informally to engage readers' interest
- **Example-presentably:** Examples are presented to clarify the equations
- **Internationally:** Measurement units are given in metric and British systems
- **Illustratively:** Technical flow diagrams improve reading ability of the reader
- **Definably:** Technical terms are defined as they are used and in a complete glossary

This book is written for the following readers:

- Beet-sugar chemists
- Beet-sugar technologists
- Sugar-technology students and instructors
- University-library audiences
- Beet-sugar researchers
- Sugarbeet farmers

Also, this book is a helpful reference for cane-sugar technologists and chemists, since some aspects of beet- and cane-sugar technology are closely related.

To Inexperienced Sugar Technologists

Mark Twain (1835–1910) in "Life on the Mississippi" states: "To make sugar is really one of the most difficult things in the world. And to make it right is next to impossible." Sugar technology may seem complicated but a user-friendly, systematic book written for the average technologist can make a big difference. Such a book was unavailable until now. This book gives special attention to the topics that often confuse beginners, offers many practical examples, and highlights important Notes to clarify the subjects and to fix concepts more firmly in your mind.

To Nonnative-English Speaking Sugar Technologists

To help readers for whom English is a second language, I have used a simple writing style to provide an easier understanding of the subjects without translation to your native language. Comparably, having this book is like having a savings account at the bank; what you put in, you get out with interest. The interest is the improvement of your English within the context of familiar subjects.

To Sugar Scientists

Standard sugar terminology (SST) does not exist for sugar technology. This often confuses the readers of sugar literature. I have tried to overcome this deficiency by choosing exact (accurate and precise) and self-defined terms (refer to Section 3 in Chapter 1), and to be consistent with the terms used in this book. Hopefully, you will find this effort to be a positive approach and will try to prepare a comprehensive SST for the sugar industry. Once a comprehensive SST is established, there will be only one exact and unified term for every concept, which can be used by people involved with the industry in all sugar-producing countries.

To Sugar Facility Directors

Hopefully, you (directors of the sugar industry, research institutes, and university departments offering major in sugar technology) will find this book to be an effective learning tool and will offer to their employees or recommend it to their students. Investing in training is an efficient strategy for managing any industrial, research, or educational facility.

To Readers

With hard work and help from a few individuals, I have written this handbook to help you (persons involved with the sugar industry). And it is finally your job to decide whether this is a **handbook** or a **shelfbook**. The ultimate test of success of any technical book is whether it will be taken in hand and used or placed on a shelf and forgotten.

Based on experience, some typing or technical errors may remain. However, they are now entirely my responsibility. Please send an email to my subject editor, Jonathan Rose, at jrose@wiley.com to let me know about any possible error or comment.

Mosen Asadi

ACKNOWLEDGMENTS

I want to express my thanks to Chris D. Rhoten for writing Chapter 7 and for his valuable technical comments on some specific subjects. Thanks also go to Jeffrey L. Carlson, PhD, for writing Chapter 10. Chris and Jeff work for Minn-Dak Farmers Cooperative Sugar Company in Wahpeton, North Dakota, and for years have been contributing to the educational side of our industry by teaching at the McGinnis Beet-Sugar Institute in Fort Collins, Colorado.

I would like to thank Lee Hubbell for reviewing Chapter 2 and Mathew Tucker for reviewing Chapter 4. Lee works as agricultural research manager and Mathew as quality manager for Michigan Sugar Company in Bay City, Michigan.

I am grateful to my daughter, Miriam Asadi, for reviewing subsection of First Aid in Chapter 11. Miriam is a PA practicing family medicine in Kalamazoo, Michigan.

Thanks also go to my friend R. Mirchi, PhD, from Prague for finding such a skilled Czech artist, Trešá Zost, to paint the images of sugar-technology masters (to whom this book is dedicated from copies of old duplicates.

Special thanks are extended to Robert L. Hetzler for his support throughout my career at Monitor Sugar Company. He was the president of the company during the years of my service to the company. What makes Bob a special individual is that he believes that "*if we are not part of the solutions, we are part of the problems.*"

At my publishing company John Wiley & Sons, Inc., Scientific, Technical, and Medical (STM) division, I want to thank Danielle Lacourciere (associate managing editor) and Jonathan Rose (associate editor) for doing their jobs in the best possible way.

Last but more importantly, my sincere appreciation goes to my family for being patient with me during four years when I was writing and fully concentrating on this book.

Mosen Asadi
Henderson, Nevada
June 2006

CHAPTER 1

BASICS OF BEET-SUGAR TECHNOLOGY

SECTIONS

1. Sugar
2. Beet-Sugar Factory
3. Sugar Terminology
4. Sucrose Properties
5. Carbohydrates

ABOUT THE CHAPTER **Chapter 1** gives all the basics related to beet-sugar technology and sugar functionalities that the readers of this book need to know to better understand the rest of the book.

Today, **beet-sugar technologists** (operators and problem solvers of the beet-sugar factories) and **sugar chemists** (operators and problem solvers of the sugar laboratories) need to know advanced beet-sugar technology, which is firmly rooted in the basics of science. The first and last chapters are complementary. The first chapter gives the basics of beet-sugar technology, and the last chapter gives the chemistry, mathematics, and statistics that relate to sugar technology. You may find it useful to refer to both chapters while studying the rest of the book.

Beet-sugar technology is the study of the production of sugar from **sugarbeet** (see the following Note) on an industrial scale. It requires a deep knowledge of two substances in sugarbeet juice:

- **Sugar** (sucrose)
- **Nonsugars** (nonsucroses)

Sugar and **nonsugars** (all soluble substances in sugarbeet juice except sugar) come to the factory in **sugarbeet** (the raw material of the beet-sugar factory). Sugar goes out as the product and nonsugars accumulate in molasses (which is a by-product of the factory). A person who has a good knowledge of sugar and nonsugars and knows how to separate them (with the highest possible efficiency) to produce sugar on an industrial scale is called a **beet-sugar technologist**.

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Beet and **cane** are almost similar in sugar content (beet typically contains 18% and cane about 15%), but are dissimilar in the amount of nonsugars (beet juice contains about 2.5% and cane juice about 5%) and fiber (beet contains about 5% and cane about 10%). The composition differences requir different methods to produce sugar from beet or cane. The differences in farming, composition, and processing of these crops are sufficient to justify two separate industries:

- Beet-sugar industry
- Cane-sugar industry

During the last century, both industries have grown considerably. World sugar production has increased from approximately 10 million tons in 1900 to about 150 million tons in 2005.

The beet-sugar industry plays an important role in the economy of beet-sugar-producing countries, which employ large numbers of people to grow sugarbeet, to produce sugar, and to support sugar-related areas such as sales, service, and research. For example, about 77000 U.S. jobs depend on beet-sugar industry. The United States produces about 30 million tons of beet/year on 0.6 million ha (1.5 million acres) of land, processes in 23 factories, and produces about 4 million tons of sugar. (There are over 500 beet-sugar factories in the world.)

About 40% of the world's sugar production is from beet, and 60% is from cane. The climates of most sugar-producing countries are suitable for growing either beet (in moderately cold areas) or cane (in tropical areas). In only a few countries (United States, Iran, Spain, Egypt, and Pakistan), the growing conditions are suitable for both crops.

Sugar from sugarbeet is produced in about 50 countries worldwide, in North America (United States and Canada), South America (Chile), Asian, North Africa (Morocco and Egypt) countries, and most of Europe.

Sugar technologists often use an old statement regarding a sugar factory's **goal**: "*The sugar factory does not make sugar; it separates nonsugars.*" But this is only half a picture because today's beet-sugar factory cannot survive if it doesn't aim for the following:

- To produce sugar with the highest efficiency
- To produce molasses with the lowest possible purity
- To be responsive to the natural environment with its wastes

Molasses desugaring by chromatographic (MDC) process has been accepted over the last two decades by the beet-sugar industry, mainly in the United States, as an efficient cost/return, environmentally safe technology. The MDC process (discussed in Chapter 8) can recover up to 90% of the sugar in molasses. A factory using the MDC process can increase the yield of **granulated-refined** (GR) **sugar** from about 80% to about 90%. This greatly improves the factory's efficiency and income, making the beet industry more secure in the market.

NOTE

Although in English-language dictionaries the term sugar beet is two words, some sugar technologists connect the words and use simply sugarbeet. This trend is followed in this book as well. The terms *sugarbeet* and *beet* are used interchangeably. Furthermore, European sugar technologists and beet growers use these terms in their singular forms (for example, % on beet), while their American counterparts use them in plural forms (e.g., % on beets). In this book, primarily their singular forms are used. For sake of comparison, we, for example, say, "some farmers grow corn," but never say, "some farmers grow corns."

CHAPTER 1

SECTION 1

SUGAR

SUBSECTIONS

- Brief History of Sugar Production
- Functionalities of Sugar
- Sweetness of Sugar
- Metabolism of Sugar in the Human Body
- Caloric Value of Sugar
- Crystal Size of Sugar
- Production and Consumption of Sugar
- Production Costs of Sugar
- Sugar Economy
- Sugar Substitutes

ABOUT THIS SECTION **This section** deals with functionalities and other subjects related to sugar. In the subsection on the economy of sugar, you will learn about major changes that the sugar industry has been going through since the last decade of the twentieth century. You will also learn about the **cooperative sugar companies** (simply, co-ops) that are owned by sugarbeet growers. The co-ops have become particularly popular in the United States in recent years.

The word **sugar** comes from the Indian *sarkara*. The chemical name of sugar is **sucrose** in English and **saccharose** in some European languages. The ***ose*** suffix (syllable attached to the end of a word) in *sucrose*, *glucose*, *raffinose*, and so on, identifies the sugars.

Sugar (sucrose, $C_{12}H_{22}O_{11}$) is one of the families of **sugars** (saccharides). All sugars belong to a larger group, known as **carbohydrates** (sugars, starches, and dietary fibers). The term **sugar substitutes** refers to all natural and synthetic (artificial) sugars other than sucrose. All sweet-taste sugars and sugar substitutes are known to us as **sweeteners**.

Sucrose (sugar), **glucose** (dextrose), and **fructose** (levulose) are examples of sweet-tasting sugars. The quantity of hydroxyl groups (OH) in molecules of sugars contributes to their sweetness. However, not all sugars are sweet in taste. In general, sugars with at least two hydroxyl groups (OH) in their molecules are sweet. About 50 compounds have a sweet taste.

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Sugar (table sugar or granulated-refined sugar) in its market-quality form is white and crystalline (granulated) with a pleasantly sweet taste. It is used in the kitchen, as an ingredient in sugar-added food products (e.g., soft drinks and confectioneries), and in production of nonfood products (e.g., detergents and ethanol). Sugar is considered:

- One of the world's purest food products (99.95% sucrose; water is the main remainder)
- One of the most natural foods used by humans for centuries without proven health risk
- One of the most purified organic compounds in the world
- One of the lowest priced food products

Beet sugar (sugar made from sugarbeet), **cane sugar** (sugar made from sugarcane), and **refined sugar** (sugar made from raw sugar) are similar in shape, taste, and other chemical and physical properties. Both beet and cane sugar (refers to their granulated refined form) that we use in cooking are pure **sucrose** with 99.95% purity, so the old belief of placing a higher value on one of them is not true. Sugar producers are not required to mention the source of the sugar on the label, so both are used interchangeably in households and in sugar-added food products.

Even experienced people find it almost impossible to recognize whether a sugar is made from sugarbeet or sugarcane. Advanced laboratory instruments and techniques are required to find the difference in beet and cane sugar by the content of **carbon isotope ratio**, which is the ratio of C^{13} (reads carbon 13) to C^{12}. This ratio is about 25% in beet sugar and 11% in cane sugar (Bubnik et al. 1995). Another differential marker is based on the raffinose content in these sugars (to a much higher extent in cane sugar) determined by chromatographic method (Eggleston 2005).

In some countries, liquid sugars (discussed in Section 20 of Chapter 3) are used by some food industries because they do not have to dissolve granulated-refined (GR) sugar before use. In a few countries (for example, the United States, Canada, and Japan), high fructose corn syrup (HFCS) has captured the liquid-sugar market because of its similarities in properties to liquid sugars and its lower production cost. HFCS is mainly used in carbonated beverage industries.

Although worldwide sugar is in a close competition with the sugar substitutes (discussed later in this section), it still has a larger market share in the sweetener industry (more than 70% of the world sweetener market belongs to sugar).

Even though sugar can be used as **added sugar** (sugar as a food additive in sugar-containing food products) in almost all food products, its properties are not entirely suitable for production of certain food products. For example, in some food products (like in fruit gums) liquid sugar may re-crystallize after the products are produced if the concentration of sugar syrup used in these products is high (more than 75%).

Before we get to other properties of sugar and basic subjects related to sugar technology, however, you might be interested briefly in knowing about the history of sugar production.

BRIEF HISTORY OF SUGAR PRODUCTION

Sugarcane cultivation and the technique of sugar production began in India probably around 2000 BC and moved to Persia (now Iran) around AD 600. In Persia, the technique was improved; milk was used as the purifying agent; and the filtered syrup was crystallized. Then

the Persians invented a cone-shaped clay mold for the production of **cone sugar** (loafsugar). The mold had a small dripping hole in the middle of its bottom so that the syrup around the crystals slowly drips from the mold. The crystals were then left to dry for few days. (Similar cone-sugar molds made of sheet metal are still used in some countries.)

In AD 800, sugarcane cultivation spread from Persia to Egypt, Syria, and as far as Morocco and Spain. By the fourteenth century, Egypt was Europe's main supplier, via the port of Alexandria, of sugar made from sugarcane.

Sugar became popular in tea in Britain by the end of the seventeenth century. In those days, sugar was available in large cone shapes that had to be broken first into large pieces with a cast-iron pincer and then into regular cube size with a little chopper.

Sugarbeet cultivation on a research scale began in 1747 when Andreas Marggraf (a German chemist) discovered sugar in sugarbeet varieties (Bruhns 1997). Later, Franz Achard (Marggraf's student) in Germany and Ya. S. Esipov in Russia were simultaneously engaged in the cultivation of sugarbeet varieties. They also continued independently with research on the processing of sugar from sugarbeet in industrial scale. The first beet-sugar factory was built in Cunern (in Germany) in 1802 by Achard and in Alyabevo (in Russia), shortly thereafter. Beet-sugar technology developed rapidly, resulting in more than 400 beet-sugar factories in European countries by 1830 (including several factories in France by the order of Napoleon Bonaparte).

The first U.S. beet-sugar factory was built in Northampton, Massachusetts, in 1838. The first two successful U.S. beet factories were built in 1870 by Dyer family in Alvarado, California, and in 1880 by Claus Spreckels in Watsonville, California. The third successful factory was built in 1890 by the Oxnard brothers in Grand Island, Nebraska (Richard 2003).

During the industrial revolution between the late eighteenth and mid-nineteenth centuries, sugar technologists had always been the models for other industries by developing new technologies and equipment.

FUNCTIONALITIES OF SUGAR

People like sugar for its sweetness. But the sweetness is only one of the **functionalities** (the factors improving the characteristics of other products) of sugar. Following are some of sugar's other functionalities:

- Improves the taste of food products
- Improves the flavor of food products
- Improves the sparkle in candy products
- Improves the shelf life of food products
- Improves the bulking property of food products
- Improves the color and texture of baked products
- Improves the preserving property of food products
- Improves the texture (mouth-feel) of food products
- Improves the foam in egg white in meringue products
- Improves the release of pectin of fruits in jam products
- Improves the heating rate of food products in the microwave
- Improves the flavor and color of food products by caramelization

- Decreases the freezing point of frozen-food products such as ice cream
- Increases the activity of yeast fermentation and the rising of baking products
- Increases the boiling-point (*BP*), allowing faster cooking at higher temperatures

NOTES

- **Low water activity in sucrose-containing food products is responsible for the freezing point depression (*FPD*) in these products. Without sugar, ice cream would freeze rock hard and would be difficult to eat.**
- **Low water activity in sucrose-containing solutions helps sugar factories by slowing rust-formation action in containers relative to containers containing just water. Generally, corrosion happens when iron reacts with oxygen in air and in water. Comparably, there are fewer free oxygen molecules in a sugar factory's juices than in water. This is the reason for less rusting activity of the factory's containers. But the headspaces of tanks and vessels not covered in the solution may suffer corrosion due to the release of volatile organic compounds (VOC) from the solution.**

SWEETNESS OF SUGAR

Sweet, salty, sour, and bitter are recognized in sensory science as basic taste feeling. Beginning in infancy, humans develop a taste for sweetness. Sweetness is sensed on the tongue and recognized by the **taste receptors** located mainly at the tip of the tongue. The receptors transfer the sweet taste through the central nervous system to the brain, which compares the sweetening power of a sweetener. The **sweetening power** depends on the following:

- Solubility of sweetener's molecules in the mouth (in water)
- Number of hydroxyl groups (OH) presence in sweetener's molecule
- Intensity of penetration of the sweetener's molecules to taste receptors
- Intensity of chemical bonding between sweetener's molecules and taste receptors

According to the **theory of sweetness**, (developed by U.S. scientist, Lamont Kier), there must be a **chemical bond** (see Glossary) between the molecules of the sweetener and the receptor to feel the sweetness. The bonds are ionic (see Chapter 12 for **ionic bonds**) between the hydroxyl group (OH) of the sweetener's molecules and the nitrogen (N) of the protein molecules in the receptor (*the better the sweet molecules fulfill the bonding requirements, the better the receptor detects the sweetness*). Sucrose, glucose, fructose, and several more compounds meet these requirements.

Sucrose (sugar) is the **standard reference** in sensory tests comparing the sweetness of other sweeteners (sugar is assigned a value of 1.0). The following list provides the approximate sweetening power of several natural and artificial sweeteners relative to sucrose, tested under the same conditions (concentration, temperature, and pH) and equal amounts. Remember that in addition to the concentration, temperature, and **pH** (measure of acidity and basicity) of the solution, the presence of other ingredients (e.g., salt) also affects the level of sweetness.

Sucrose	1.0
Glucose	0.7
Fructose	1.0
HFCS*	1.0

Invert sugar	1.2
Acesulfame	150
Aspartame	180
Saccharin	300
Sucralose	600

*High-fructose corn syrup

As you can see, some sweeteners (mainly artificial sweeteners) are sweeter than sucrose. For example, sucralose (see Notes) is an artificial sweetener, which is 600 times sweeter than sucrose. Sucralose (its brand name is Splenda) is a chlorinated compound, and its molecules correspond to those of sucrose, but three of its hydroxyl groups are replaced by chlorine (Cl), which creates an artificial taste compared with natural sugar.

NOTES

- **The high sweetness of sucralose was discovered when a researcher misunderstood a supervisor's request to *test* a new chlorinated compound, and instead *tasted* it.**
- **The word *ose* in sucralose does not include it in the natural sugar group.**

METABOLISM OF SUGAR IN THE HUMAN BODY

Sucrose is a simple carbohydrate with a fast metabolism (digestibility characteristic). In the digestive system, an **enzyme** called **sucrase** (see the following Notes) converts the sucrose to glucose and fructose.

$$C_{12}H_{22}O_{11} + H_2O + \text{Enzyme} \rightarrow C_6H_{12}O_6 + C_6H_{12}O_6$$

In the liver, the fructose is converted to its **isomer** (compounds with the same chemical formula but different structures), **glucose** (known as **blood sugar**), to be used in the blood as the source of energy. Blood carries oxygen (O_2), absorbed from the air by inhalation, to the various cells of the body. The cells oxidize the glucose by combining it with the oxygen. The **oxidation** (simply, reaction of a substance with oxygen) of glucose produces considerable amounts of energy:

$$C_6H_{12}O_6 + 6O_2 \rightarrow 6CO_2 + 6H_2O + \text{Energy}$$

This reaction is an **exothermic reaction** (energy-releasing reaction), releasing 2800 kJ (669 kcal) energy per mole of $C_6H_{12}O_6$ (or 669/180 = 3.7 kcal/g). (The value 180 is the glucose molecular mass.) The released energy enters the bloodstream to be used as the source of energy. This is a fast reaction, so it is used when a fast response to energy deficiency is needed. In hospitals, the glucose solution is injected directly into a patient's vein to restore the blood glucose level. We also know that athletes drink sweet beverages before sporting events.

In the body, all carbohydrates (sugars, starches, and dietary fibers) eventually convert to glucose to produce energy. The energy required to perform individual functions by the body is much less than the energy produced by glucose conversion. In our body, about half of the energy produced by glucose conversion is used to do activities. The remainder of the energy is released as heat to maintain the body's temperature. In addition, part of glucose is converted to **glycogen** (a polysaccharide) and stored in the liver and muscles to be used later. When the body needs energy, glycogen is broken down again into glucose to maintain blood glucose level.

The normal range of glucose in an adult's blood is between 70 to 100 mg per 100 mL (0.07 to 0.10%).

Diabetes is a disease in which the body either does not produce **insulin** (a hormone that regulates glucose level in the blood) or does not use it properly to allow glucose (blood sugar) to be converted to energy. So the extra glucose builds up in the blood causing a high glucose level, known as a high blood-sugar level.

NOTES

- **Although the term diabetes applies to the high level of glucose (as the product of all carbohydrates in the body), sucrose (sugar) is the only carbohydrate that gets the blame for diabetes, and just because of the generality of the word "sugar" in peoples' minds.**
- **The competitors of the sugar industry try to make sugar a label-unfriendly ingredient and mislead people that consumption of sugar (sucrose) is the cause of diseases such as diabetes, heart problems, and hyperactivity in children, but with no accepted scientific proof. A connection with some diseases exists but by way of the overconsumption of daily caloric intake, which creates obesity. The majority of our daily caloric intake comes from other sources (fats, proteins, and starches) than sugar. So far, scientists have confirmed no health risks related to a moderate use of sugar.**
- **In enzyme chemistry, the names of enzymes often end with *ase* and often describe the compound on which an enzyme works. For example, the enzyme sucrase hydrolyzes sucrose.**

CALORIC VALUE OF SUGAR

A **calorie** (cal) is the amount of energy required to raise the temperature of 1 g of water 1°C (actually from 15 to 16°C). The **kilocalorie** (kcal, Cal or Calorie with capital C), equivalent to 1000 cal, is used as the **dietary** (food) **calorie**, which expresses the nutritional value of food products. (Note: Most of the time calorie [cal] is simply referred to as a Calorie or **caloric value**.)

The **serving size** (the base for reporting each food) of sugar is *one teaspoon (4 g)*, which contains 16 Calories (16 kcal) or 66 kilojoule (**kJ**, joule rhymes with *tool*) of nutritional energy. In the United States, the FDA requires that the serving size be expressed in both common household terms and metric units (e.g., one teaspoon, 4 g). For detailed calculations of the amount of calories of sucrose, refer to the Sucrose Specific Enthalpy subsection in Section 4 of this chapter.

An equation, the "4-4-9 equation" is used to calculate the caloric value of food products. To find the amount of calories in a food product, the following are determined:

Amount of fat in the product
Amount of protein
Amount of water
Amount of ash
Total of above

$$\text{Amount of carbohydrate} = \text{Actual weight} - \text{Total of above}$$

$$\text{Calories} = (\text{g carbohydrate} \times 4) + (\text{g protein} \times 4) + (\text{g fat} \times 9)$$

The 4-4-9 equation is based on:

Carbohydrates	4 Calories/g
Proteins	4 Calories/g
Fats	9 Calories/g

Human caloric requirements depend on:

- Age
- Body size
- Metabolic rate
- Physical activity

A rough estimate of a minimum caloric intake to maintain body activity is about 1 Cal/kg.h (1 Cal per each kg of body mass per hour). Average daily human caloric intake varies with age:

About 700 Calories for a 3-year-old baby
About 1500 Calories for a 25-year-old woman
About 2000 Calories for a 25-year-old man

The FDA determines the daily food intake based on 2000 Calories. The **average daily intake** (ADI) of all sweeteners in the United States in 2005 was about 125 g per day, mainly used in sweetening drinks. This equates to 480 Calories, or about 25% of the **daily calorie intake** (based on 2000 Calories per day). Figures 1.1 and 1.2 show the daily sweetener consumption and calorie intake from sweeteners in the United States from 1985 to 2005 (USDA 2005).

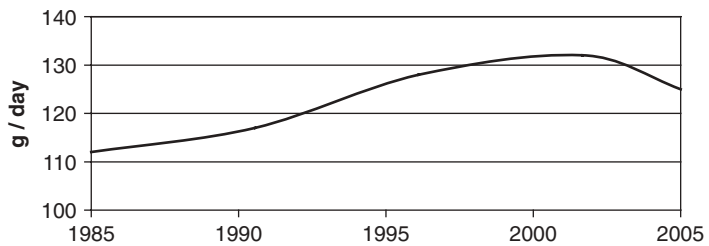


Figure 1.1 Daily sweetener consumption in the United States since 1985

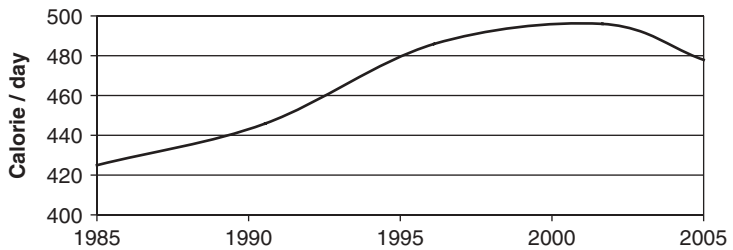


Figure 1.2 Daily calorie intake from sweeteners in the United States since 1985

NOTES

- **Sugar is a luxury food in some less developed countries, where people are short of calories (an unfortunate fact), while in developed countries some people try to consume less sugar because of its calories.**
- **World per-capita sugar consumption increased from about 220 Calories/day in 1985 to 280 in 2005. This equates to about 55 g/day (20 kg/year) in 1985 and 70 g (25 kg/year) in 2005.**

CRYSTAL SIZE OF SUGAR

Sugar factories in different countries produce sugar of certain crystal sizes, depending on the habits of the household and commercial use. In general, household consumers in the United States prefer fine sugar, those in Europe prefer medium-size sugar, and those in Asia, Africa, and the Far East prefer coarse sugar.

The size of sugar crystals is expressed by the **mean aperture** (*MA*). The *MA* is the average crystal size of the sugar, expressed as the size of the screen in millimeter (mm) or in micrometer (μm) on which 50% of the sugar sample stays and 50% passes through. Besides the crystal size, the **uniformity** (distribution) of the crystals is also important. The uniformity of the sugar is expressed by its **coefficient of variation** (*CV*). (For more information on *MA* and *CV*, see Chapter 4.) Standard crystal sizes are as follows:

- Extra-fine sugar with an *MA* of about 200 μm (0.2 mm)
- Fine sugar with an *MA* of about 300 μm
- Medium-size sugar with an *MA* of about 500 μm
- Coarse sugar with an *MA* of about 1000 μm
- Extra-coarse sugar with an *MA* above 1000 μm

For example, the baking industry uses extra-fine sugar because it mixes well during processing (due to its high surface area) and produces dough with higher rising property, and finely baked products with better quality (finer texture and less crumbs).

In most countries, fine and medium-size sugars are usually the standard, and extra-fine, coarse, and extra-coarse sugars are considered **specialty sugars** (see Section 20 in Chapter 3), for which customers pay a premium.

PRODUCTION AND CONSUMPTION OF SUGAR

World sugar production (from beet and cane) in 2005 was 149.5 million **metric tons raw value** (MTRV) and consumption was 150.5 MTRV. (Sugar production and consumption is usually expressed in raw sugar equivalent MTRV.) World sugar consumption has grown by 3% annually since 1985, and the world production has kept up with this increase in demand for sugar. Figure 1.3 shows world sugar production since 1985.

The 25 member countries of the **European Union** (EU-25) are the world's third-largest sugar producers (after Brazil and India) with about 16 million tons sugar production and the second-largest exporter of sugar (after Brazil) with about 6 million tons sugar export. Twenty-one countries of the EU-25 are sugarbeet-growing countries.

In the United States in 2005, the sugar production from sugarbeet was about 4 million tons raw value and from cane was 3.9 million tons. The U.S. production from sugarbeet has

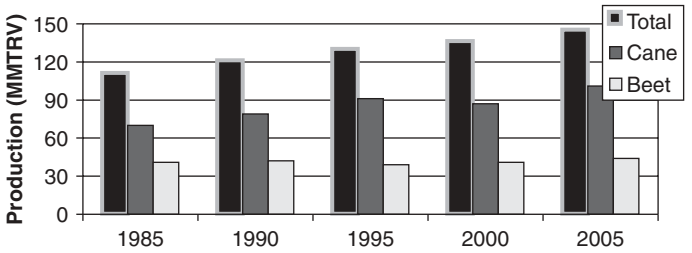


Figure 1.3 World sugar production

been essentially around 4 million tons since 1990. What has changed is that fewer factories are producing sugar (there were 36 beet factiories in the United States in 1990 and 23 in 2005).

In recent years, world sugar **consumption** has increased almost yearly, to about 25 kg (55 lb) per capita per year in 2005. This includes household consumption and added sugar (sugar as a food additive in sugar-containing food products such as beverages and confectioneries). Household consumption accounts for almost one-third of the total consumption. It is estimated that the world consumption per capita will be 30 kg (66 lb) in 2010. Nevertheless, sugar consumption has decreased in some countries. For example, in the United States, the per capita intake has decreased from 29 kg (64 lb) in 1990 to 28 kg (61 lb) or about 77 g/day in 2005.

The following list shows the sugar production and consumption (capita intake) in 2005 in three countries with the highest sugar production in the world.

	Production (M tons)	Consumption (kg/capita)
Brazil	28.2	60
India	19.1	19
EU	16.7	42
World	149.5	25

NOTES

- **The United States and India combined consume 46 million tons of sugar a year, making them the largest consumers of sugar in the world. This equates to 30% of the world's consumption. China is next, with about 10 million tons consumption a year. Consumption in China is projected to increase to 15 million tons in 2010 (Bouvier 2005).**
- **U.S. per capita consumption of sweeteners is almost 3 times higher than India and 10 times higher than China.**
- **In the world sugar market, the base (raw sugar with 96% sucrose) is used for expressing sugar production as raw value (*RV*). Usually granulated-refined (GR) sugar and liquid-sugar value are converted to raw value based on the percentage of sugar content (*S*), measured by polarimeter (called polarimetric sugar or pol) by using the following formula:**

$$RV = 100 \times [(1.07 - (100 - S) \times 0.0175] \quad (1.1)$$

EXAMPLE 1

Given: 100 kg raw sugar with 97.7% sugar content
Calculate: *RV*

$$RV = 100 \times [(1.07 - (100 - 97.7) \times 0.0175] = 103.0\,\text{kg}$$

This means that the sugar refinery makes *about* 100 kg of granulated-refined sugar from processing 103 kg of this raw sugar. Refer to Example 2 in Chapter 9 for more information.

PRODUCTION COSTS OF SUGAR

Sugar is produced from two different raw materials: **sugarbeet** and **sugarcane**, which are grown in different geographical areas with different production costs. The production costs of sugar consist of the following:

- **Labor costs**: Wages, benefits, and health insurance
- **Raw material costs**: Sugarbeet or sugarcane growing costs
- **Processing costs**: Fuel, chemicals, maintenance, and other related costs

These costs are different depending on which raw material is used. Most importantly, in the cane-sugar factories, **bagasse** (desugared-fibrous residue left after milling sugarcane) is used as fuel in boilers for the production of steam. (In some cases, cane factories even produce energy surplus, which is sold to public system.) The desugared-fibrous residue left after sugarbeet processing, called **pulp**, cannot burn properly, so beet-sugar factories do not have a suitable by-product to use as source of energy for boilers. In addition, the production costs of sugarbeet and sugarcane in the field is different. Such dissimilarities create a considerable difference in production costs of sugar from sugarbeet and sugarcane in the field as well as at the factory levels. But there is no difference in sugar price when it gets to the market, since both crops produce the same product.

Although transportation costs are incurred in cane-sugar production, because cane raw sugar is usually produced in a cane factory and then refined to granulated-refined (GR) sugar at a sugar refinery, the *production costs of cane sugar are lower than that of beet sugar.* Investigations have indicated that the **weighted average** (the average that gives points to the priorities–here, the average that gives points to countries with higher production) of world production costs, at both the field and the factory levels, is substantially lower (by about 60%) in favor of cane-sugar producers (Kingsman 2002).

The difference in production costs puts a great challenge on beet-sugar producers to be *highly efficient*. Some countries have met this challenge. For example, despite high labor and environmental protection costs, the U.S. beet-sugar producers have among the world's lowest production costs (despite high labor costs) because of high efficiency and the use of the **molasses desugaring by chromatographic** (MDC) **process**. Chile is another low-cost producer of beet sugar. Ukraine (with about 100 beet-sugar factories) is the highest cost producer, well above the average world production cost. In cane-sugar production, Brazil, Australia, and South Africa produce sugar below the average world production cost (Kingsman 2002).

Raw material costs account for a large part of total production costs. Factories pay farmers for tons of delivered sugarbeet. In many countries, the *sucrose content* also affects the price of the sugarbeet delivered to the processor. High-sucrose and low-nonsucrose content are the most important factors affecting the quality of the sugarbeet. Sugarbeet production costs in the field

vary considerably among countries. In 2000, the production cost of sugarbeet in the United States ranged from $15.40 to more than $60.00 per ton, with the average of $37.30 (USDA 2004). (Note: The production costs are higher in western parts of the United States because of the cost of irrigation.)

SUGAR ECONOMY

From 1990 until late 2004, the sugar industry experienced a decrease in sugar prices. During this period, sometimes the price of sugar fell even below production costs, considerably affecting the profitability of sugar companies. Flat or declining sugar prices in those years led to problems in some producing countries where rising production costs were not offset by profit.

The main reasons for the decrease in sugar prices in those years are the following:

- Barrier international sugar regulations on price stabilization
- Changes in sugar tariffs in different countries
- Competition with sugar substitutes in some countries
- Dependability of the market on world sugar surplus and stock
- Reliability of the market on the regulation of duty-free import quotas
- Decrease in governments buying sugar during buyer shortages (Pack 2003)
- Reliability of the market on Brazil as the world's primary sugar producer and exporter and Brazil's interest in lowering the world price of sugar due to the devaluation of its currency (Chavanes 2003)

In addition, the world price of sugar depends on oil price. Furthermore, it also depends on weather, economic and political stability of the individual countries and their desire to produce sugar.

Since early 2005, the world price of sugar has started to rise, reaching an increase of 35% by the end of that year. It is expected that sugar price will keep its rising trend during 2006, reaching a quartercentury high. Besides the increase in oil price and its connection to sugar price, the other main reasons for the price rise of sugar are the following:

- Brazil has started to use more of its crop to produce ethanol due to recent high oil price
- World consumption has increased (from about 146 M tons in 2004 to about 150 Mt in 2005)
- World stocks of sugar have recently dropped (from about 35 Mt in 2004 to about 31 Mt in 2005), e.g., U.S. stock-to-use ratio in 2004 was about 18.8% and dropped to 12.8% in 2005

Despite fall and rise in sugar prices since 1990, production costs of sugar have increased steadily during this time because of the following reasons:

- Increase in oil price
- Increase in labor cost
- Increase in equipment cost
- Increase in barriers in environmental policies
- Decrease in funds for factory maintenance and improvement
- Decrease in national subsidies (sugar-price support and loan programs by some governments)

Knowing that sugar is a politically involved commodity and governments can never satisfy all involved parties, the primary responsibility of the sugar industry is to produce sugar at the lowest cost possible. To do so, the industry has decided to move toward the following three main goals:

- Improve the type of management and ownership of sugar companies
- Use new cost-effective technologies and cost-saving measures
- Adapt to national and international sugar regulations

On the side of improvement in the type of management and ownership, the beet-sugar industry has initiated important policies during the last two decades to become more competitive. Among other plans, the two most successful policies instituted by the industry are the following:

- Managing sugar companies as cooperatives
- Merging sugar companies

Cooperative sugar companies (simply, co-ops) were established in the United States to manage the production facilities more efficiently. Beet-sugar co-ops are owned by growers and are performing considerably well. In the United States, the first beet-sugar co-op was formed in 1899 in California. In 1973, the first large beet-sugar company of this type was formed when sugarbeet growers purchased the American Crystal Sugar Company in North Dakota. This trend continued with three new factories built in North Dakota and Minnesota. In 1997, sugarbeet growers started to negotiate over the financial assets of the Amalgamated Sugar Company and succeeded later to purchase the company. In 2002, the Michigan Sugar Company (with four sugar factories) was bought by sugarbeet farmers. In late 2004, the Monitor Sugar Company (with one large factory) was the latest company bought by local beet growers and merged into the Michigan Sugar Company.

In the United States in 2005, all 9 beet-sugar companies (with 23 factories) were co-ops, accounting for 100% of the total processing capacity (165000 t/day in all factories).

In Europe, co-ops are also increasing in popularity. Today, growers are the main shareholders of some larger European sugar companies. For example, in 2005, growers held 56% of the shares of Südzucker in Germany.

The major advantages of co-ops compared with privately owned or investor-type companies are as follows:

- **Increased efficiency and profitability**: In a co-op operation, owners (beet growers) are interested in the efficiency of the factory as well as in the improvement of the quality of the sugarbeet. (Co-ops have been more successful than privately owned companies in producing quality sugarbeet.) Growers in charge of both important operations can increase profits to shareholders. In other words, the profit goes to the growers in a co-op, but it is shared among growers and the processor in a privately owned company.
- **Tax advantages**: The sugar factory does not produce a profit and thus pays no taxes based on U.S. tax regulations. All profits from the business are passed on to the growers except those used to pay debts or to make capital improvements to maintain or raise plant efficiency and capacity. Growers pay taxes on their income, but the processing facility is converted to a cost-saving center in the overall production of the sugar. As a result, the grower receives the maximum levels of income possible from sugarbeet production. Capital invest-

ment for raising the factory's efficiency or reducing operating costs becomes a benefit to the grower.

The major disadvantage of co-ops is that these companies usually have a high debt load from the factory buyout. At least until they can reduce their debt, they are financially constrained when it comes to maintaining and upgrading their factories properly.

Merging (consolidating) **sugar companies** and operating them under joint management in the form of a **multiple-factory company** is another way of decreasing expenses and increasing income. Merging shareholder-owned sugar companies has been particularly the hallmark of Western European countries since the 1990s revolution in Eastern European countries. Since then, the Western sugar companies have bought several eastern-European factories, closed some of them, and reconstructed others to more modern factories with high operating capacity and well-established income.

On the side of using new cost-effective technologies and cost-saving policies, the sugar industry aims toward the following goals:

- Increase the profitability by using new technologies (e.g., molasses-desugaring process)
- Use energy-saving equipment such as steam-pulp dryers and plate evaporators
- Increase the efficiency of the factories and decrease processing sugar loss
- Make equipment perform more than one duty (e.g., refining raw-cane sugar)
- Increase the lifetime of equipment by proper maintenance
- Increase the efficiency of the sugarbeet fields
- Increase the capacity of the factories
- Decrease workforce

Molasses desugaring by chromatographic (MDC) process is a new technology, capable of recovering up to 90% of the sugar in molasses. This increases the yield of sugar from beet from about 80% in a conventional beet factory to about 90% in a factory equipped with the MDC technology. This considerably improves the income and makes the beet-sugar industry more secure in the world sugar market. In 2006, 8 of the 23 American beet-sugar factories were equipped with the MDC process. Five of eight American beet-sugar companies are multimember factories (each company has two to five factories) and each company has equipped at least one of its factories with the MDC technology.

With the increase in fuel costs since 2002, much effort has been directed toward the creation of a more **energy-efficient** sugar factory. The sugar industry is a **high-energy user**, so energy-saving equipment such as steam-pulp drying and plate evaporators are important examples of new technologies used in recent years in beet-sugar factories to improve the efficiency and return on investment (ROI) of newly built and expanded factories.

Factory capacity (amount of beets processed in 24 hours) in many countries, such as France, Germany, and the United States, has been increased to improve the efficiency of the factories. For example, in 1980, the United States had 53 beet-sugar factories, with an average capacity of about 3000 t/day; in 1990, there were 36 factories, with an average capacity of 4700 t/day. In 2004, there were only 25 factories, with an average capacity of about 7000 t/day. In 2005, Amalgamated Sugar stopped the sugarbeet operation in its Nyssa factory, and Michigan Sugar discontinued the operation in its Carrolton factory. The Latest shutdowns reduced

the number of U.S. beet factories to 23 and increased the average size of their operations to about 7200 t/day. As a result, fewer producers are left who produce more sugar at a lower profit per unit of production.

Canada also has recently closed a few of its factories (mainly because of difficult domestic sugar regulations). In 2005, Canada had only one sugarbeet-processing facility (Rogers factory) at Taber, Alberta (operated under Rogers Sugar Ltd.) with a capacity of about 6000 t/day. However, Canadian sugarbeet growers located in the southwestern of Ontario send their sugarbeet across the border to be processed at the Croswell factory (one of the Michigan Sugar factories).

The sugar industry has high capital costs due to the use of heavg equipment. In general, the required capital cost to install a new beet-sugar factory (a conventional factory without the MDC process) ranges from $15000 to $25000 per ton of daily beet processing. The capital cost is higher by about 25% for expansion and reconstruction of old factories (Van der Poel 1998). The reconstruction of a sugar factory costs between $10 and $50 million, depending on the equipment the factory already has in place (Rawlings 1998).

Today's beet-sugar factories have sufficiently large processing capacity (6000 to 24,000 tons per day) to achieve reasonable returns on investment (ROI). The ROI for a newly built sugar factory ranges from 15 to 20 years, depending on the capacity of the factory, the length of the beet campaign, and the lifetime of the factory equipment. The **lifetime** of sugar factory equipment differs from country to country (van der Poel et al. 1998). For example, in Germany, the lifetime for different equipment is estimated as follows:

Boiler	15 years
Evaporator	12 years
Heater	20 years
Pan crystallizer	12 years
Building	50 years

In the United States, the equipment lifetime is about triple these estimates. With recent changes in EU sugar reform in 2006, the Europeans will also have to make their equipment last longer because they simply will not have the capital to replace equipment at the present rate.

The sugar industry tries to adapt national regulations (e.g., revision of the U.S. Farm Act in 2007) and international regulations (e.g., revision of the EU sugar regime in 2006) to comply with the WTO (World Trade Organization) regulations towards maintaining a reasonable and stable sugar price. Mainly, the sugar industry cooperates with intergovernmental bodies, such as the WTO and the ISO (the International Sugar Organization), to convince influential people involved in the decision making of sugar polices in different countries to deal globally with sugar trade, tariffs, and price stabilization. Global trade liberalization based on an open-market policy not only benefits sugar producers but also consumers. The EU sugar price cut in 2006, the elimination of agricultural export subsidies by 2008, and the provision of duty-free/quota-free market access from the 32 least-developed countries by 2013 are examples of important decisions that were or will be dealt with in the near future.

Sugar producers are not the only ones who pay for the fluctuation of world sugar prices; **household users** (as the end-consumers) pay as well. This is because **industrial users** of sugar adjust the price of their products only when the price of sugar goes up. (Note: About two-thirds

of the sugar in the world is used industrially as added sugar, for example, in the confectionery products.)

In a research study among developed countries in 2000, consumers in Norway paid the highest ($1.90 per kg or 86 cents per pound) and consumers in Australia paid the lowest retail price for sugar (73 cents per kg or 33 cents per pound). Americans consumers paid 95 cents per kg (43 cents per pound), which is 20% below the average price paid in other developed countries.

Overall, the sugar industry is optimistic for a better future for the following reasons:

- Rise in international demand for sugar
- Rise in international demand for ethanol-blended fuel
- Recent price adjustment stabilized on a global scale
- Much larger market share of sugar among sweetener industries (more than 70% of the world sweetener market belongs to sugar)

However, the sugar industry is well aware of the fact that unreasonably high prices promote the growth of its main competitors, the **sugar-substitute** industries.

SUGAR SUBSTITUTES

Sugar substitutes (sugarless sweeteners or sugar replacers) are alternative sweeteners with less sugar-like taste. Over the last century, particularly since the 1980s, several artificial and natural substitutes have been offered to consumers to compete with sugar. Some sweeteners are many times sweeter and less caloric (provide fewer calories) than sugar, so smaller quantities are needed to sweeten food. The sugar substitute were developed for four main reasons:

- Lower calories
- Diabetic consumption
- Lower production costs
- Higher sweetening power

Sugar substitutes can be divided into two groups:

- **Artificial sugar substitutes:** Acesulfame-K, aspartame (trade name, NutraSweet and Equal), aspartame-acesulfame salt (Twin-sweet), and saccharin ($C_7H_5NO_3S$, SweetNLow), sucralose (Splenda), neotame, and alitame are examples of artificial sweeteners.
- **Natural sugar substitutes:** High-fructose corn syrup (HFCS), glucose, and fructose are the main natural sugar substitutes.

HFCS is a natural sweetener produced by **hydrolysis** (the reaction of a compound with water) of **starch** (a polysaccharide containing many glucose units). The starch is hydrolyzed to glucose; then part of the glucose is enzymatically converted to fructose. Corn is the most economical and abundant source of starch for production of HFCS in many countries. It was introduced by the U.S. corn industry in 1967. HFCS is particularly used in carbonated beverages. Since 1985, 55% HFCS has been the main source of sugar in carbonated beverages in the United States and the main competitor of sugar, because of lower production costs of about 15%. Figure 1.4 compares consumption of sugar with HFCS.

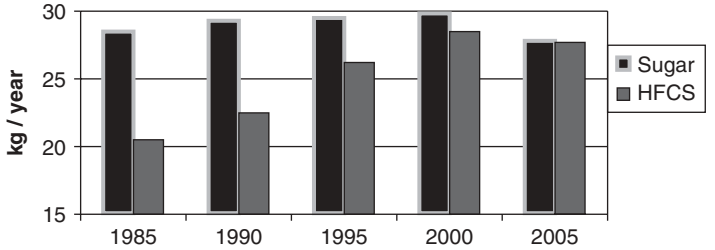


Figure 1.4 Sugar and HFCS consumption in the United States since 1985

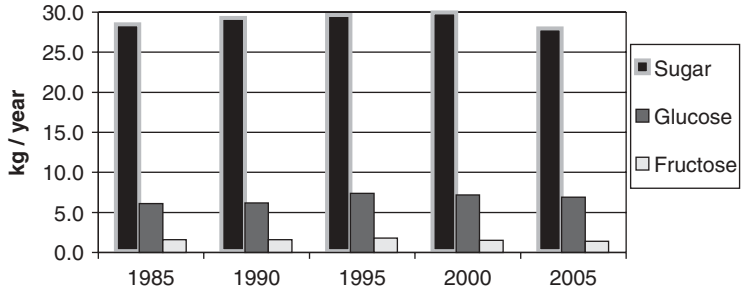


Figure 1.5 Sugar, glucose, and fructose consumption in the United States since 1985

Unlike HFCS, crystalline glucose and fructose are more expensive to produce and cannot match sugar in functionality; therefore, they cannot compete effectively with sugar. Figure 1.5 compares consumption of sugar with glucose and fructose in the United States since 1985.

Neohesperidin dihydrochalcone (**NHDC**) is an intense and nonnutritive sugar substitute. NHDC is produced from the peel and skin of citrus fruits. Added at low concentrations, it can contribute to sweetness. **Flavonoids** (flavones, flavonols, and anthocyanins) are subgroups of the NHDC. Flavonoids are dietary compounds that have **antioxidant** properties (see the Note below). They are found in tea, vegetables, fruits, and red wine.

Glycyrrhizin (extracted from licorice root), stevioside (extracted from South American plant leaf), and thaumatin (extracted from an African fruit) are some other intensive sweeteners, about 50, 200, and 3500 times sweeter than sucrose.

In general, sugar substitutes cannot create the *exact functionalities* in food products as sugar does. Some substitutes, such as aspartame, cannot be used directly for cooking because they decompose at high temperatures. In addition, aspartame creates a health hazard to persons who cannot properly metabolize phenylalanine (phenylketonurics). Therefore, the FDA requires a "Contains phenylalanine" warning on the label of foods that contain aspartame.

NOTES

- **Anioxidants (mainly vitamins A, C, D, E, and K) are substances that block the oxidation process of free radicals. Free radicals are produced in our body during digestion of foods and are highly reactive atoms that lack an electron. Antioxidants give the extra electron to free radicals, thus preventing the electron from being taken from a healthy cell. Free radicals are destructive to healthy cells and DNA (deoxyribonucleic acid, the substance that causes a cell to divide and reproduce)**

because they must take an electron from a cell to stabilize. In our body, free radicals act like oxidants, and so the vitamins that prevent their action are called antioxidants. Factors, such as aging, pollution, tobacco, radioactivity, and some medical conditions increase free-radical formation. Our body cannot produce antioxidants but can get them from food (mainly fruits) or vitamin supplements.

- **Today, some people prefer sugar-free products (particularly carbonated beverages made from artificial sweeteners) to reduce the amount of calories in their diets. However, consuming extra chemicals (particularly low-calorie products whose side effects are not investigated) can be a health risk. Furthermore, some sugar substitutes have not yet investigated for toxicity (poisonous ability), carcinogenicity (cancer-causing ability), and other disease tests.**
- **Products containing sugar substitutes are usually labeled sugar free to suggest "calorie free" to consumers, which is not a true statement.**

CHAPTER
1

SECTION 2

BEET-SUGAR FACTORY

SUBSECTIONS

- Beet-End and Sugar-End
- Campaign and Intercampaign
- Sugar Extraction and Recovery
- By-products of the Beet-Sugar Factory
- Managerial Structure and Ownership

ABOUT THIS SECTION **This section** familiarizes the reader with the beet-sugar factory and its product and by-products. In addition, it discusses the organization of the beet-sugar factories.

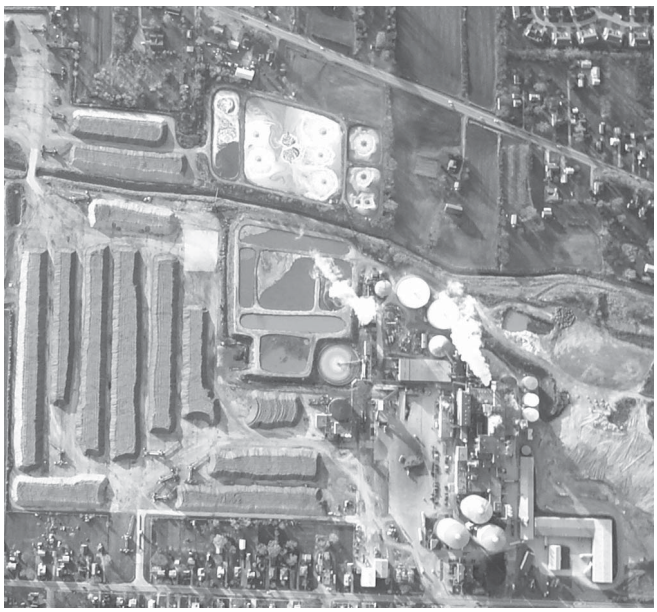


Figure 1.6 A beet-sugar factory

Beet-Sugar Handbook, by Mosen Asadi
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The **beet-sugar factory** is a facility for recovering and refining sugar from sugarbeet. As stated earlier, the main goal of the sugar factory is to separate nonsugars (unwanted components) from sugar to produce granulated-refined (GR) sugar with the highest possible efficiency *(the sugar factory must not only be operable but also efficient)*.

In the beet factory, the separation of sugar (sucrose) from nonsugars (nonsucroses) takes place in different stages, known as **stations**. The **diffusion station** is the starting point of this separation. It is followed by juice purification and crystallization. Sucrose is not the only substance in the diffusion process subject to movement from sugarbeet cells; nonsucroses are also moved (unintentionally) to produce an impure sugar, solution known as **diffusion juice** (raw juice). The diffusion juice contains approximately 15% dry substance (*DS*) and 85% water. Out of 15% *DS*, about 13% is sucrose (*S*) and 2% is nonsucroses (*NS*). The **purity** (*P*, sucrose content as % of dry substance) of this juice can be determined by the purity equation:

$$P = \frac{S}{DS} \times 100$$
$$P = \frac{13}{15} \times 100 = 86.7\% \tag{1.2}$$

From the diffusion stage onward, the processing goal is to improve the juice's purity to the extent that sugar with 99.9% purity is produced.

Sugar factories (beet and cane) can be divided into the following four types, based on the raw material (sugarbeet, sugarcane, or raw sugar):

- **Beet-sugar factory:** Produces raw sugar from sugarbeet and processes it into refined sugar
- **Cane-sugar factory:** Produces raw sugar from sugarcane and processes it into refined sugar
- **Sugar mill:** Produces raw cane sugar from sugarcane and ships to sugar refineries for refining
- **Sugar refinery:** Produces refined sugar from raw cane sugar (refineries are usually located close to a waterway to receive raw sugar transported by ship)

Following are the major differences between a beet factory and a cane factory or a sugar mill:

- Beet factories do not have a suitable by-product to use as fuel for boilers (the pulp cannot burn properly), but cane factories and sugar mills use **bagasse** (cane fiber) as boilers' fuel.
- Beet factories have a limekiln to produce their lime requirement, but cane factories and sugar mills purchase lime because they require much less lime than beet factories.
- Beet factories use the complete-liming process to destroy **invert sugar** (a mixture of equal parts of glucose and fructose), but cane factories, and sugar mills apply partial liming (part of invert sugar stays in molasses).
- Beet factories produce molasses with about 60% purity (with low invert sugar), but cane factories and sugar mills produce molasses with about 40% purity (with high invert sugar).

Factory capacity (also known as **slicing rate** or throughput of the factory) expresses the size of the sugar factory with regard to its ability to process certain tons of sugarbeets in a 24-hour period. Today, beet-sugar factories are large. Their **processing capacities** range from 2000 to

20000 tons of sugarbeets, and thus they produce about 300 to 3000 t of sugar per day. (Note: France has the largest beet-sugar factories in the world, with capacities up to 24000 t/day.)

As of 2006, there are about 500 beet-sugar factories worldwide, with a majority in Europe (about 300). There are also beet-sugar factories in Asia (about 100), North Africa (about 15), North America (24), and South America (5). In Europe, Ukraine has the highest number of sugar factories (about 100). In Asia, Iran is the country with the highest number of beet-sugar factories (Iran has 35 beet and 2 cane factories). (Note that these numbers may fluctuate with the installation of new factories or the closure of old ones, increasing the capacity of the remaining factories.)

Following are the four types of beet-sugar factory:

- **Beet-sugar factory:** Produces raw sugar from beet and refines it to produce refined sugar.
- **Beet-sugar factory with molasses-desugaring process:** Produces sugar from beet and desugars molasses by the molasses-desugaring process.
- **Beet-sugar factory with raw cane sugar refining:** Produces sugar from beet and refines the raw cane sugar during the beet campaign or intercampaign (few factories are of this type).
- **Raw factory:** Produces either raw beet sugar or thick juice to be shipped to another factory for final processing into refined sugar (few, if any, factories are of this type).

NOTE

In this book, the term sugar factory denotes a conventional beet-sugar factory (the first type of the groups listed above) without the desugaring process or raw cane sugar processing.

BEET-END AND SUGAR-END

North American beet-sugar technologists divide the sugar factory into two main sections:

- Beet-end
- Sugar-end

The **beet-end** of the factory includes the processing steps up through the thin-juice evaporation station and the production of thick juice. The **sugar-end** consists of crystallization, centrifugation, sugar conditioning, sugar silos, the sugar warehouse, and production of specialty sugars. The duty of the sugar-end is to crystallize sucrose present in the thick juice to granulated-refined sugar (product of the sugar-end) and molasses (by-product of the sugar-end). Because no nonsugars are eliminated in sugar-end operation, except in the molasses (disregarding losses), the amount of sugar recovered as granulated-refined (GR) sugar and the amount of sugar left in the molasses depend entirely on the purity of the molasses produced.

European beet-sugar technologists usually divide the sugar factory into two sections:

- **Raw-side**: From beet washing up to the production of raw sugar
- **Refinery-side** (sugar house): Production of refined sugar

In some factories, the beet-end is built to process more sugarbeet than the sugar-end can handle. In these factories, thick juice or standard liquor is concentrated and stored in storage tanks for processing during the **juice-campaign** operation (see Section 15 in Chapter 3). It is important

to point out that during the juice campaign, mainly the sugar-end section of the factory, the boiler house, and the evaporation station are in operation.

NOTE

Beet-end operations eliminate some impurities, but the sugar-end is where the job is finished by the complete separation of impurities and the production of granulated-refined (GR) sugar, which is pure sucrose.

CAMPAIGN AND INTERCAMPAIGN

The **beet campaign** (beet-processing period) lasts 3 to 7 months, depending on sugarbeet availability and weather conditions. Usually, it starts in mid-September and continues until late February, or even until mid-April in some areas. The beet campaign lasts 3 to 4 months in Europe, and up to 6 months in the United States. An exception to this schedule occurs in the central valley of California (as well as in other warm areas around the world), where the slicing season is 8 to 9 months, from late March through November.

The beet-processing period is the most important time for the sugar factory. When the beet processing starts, the factory works 24 hours a day, 7 days a week, until all of the sugarbeets are processed. During the campaign period, breakdowns are the only reason for a shutdown of a section of the factory.

During the **intercampaign** (off-beet campaign), the equipment is maintained to prepare the factory for the next campaign. Production lines on **specialty sugars** such as powdered, brown, and cube sugars continue their activities using the granulated-refined sugar produced earlier and stored in sugar silos. During the intercampaign, the sugar-end of the factory processes thick juice for storage (if factory is equipped with thick juice storage) or extract (if factory is equipped with the desugaring process by chromatographic process). The factory also continues selling sugar in bulk.

SUGAR EXTRACTION AND RECOVERY

Sugar extraction (E, sugar yield) is a percentage ratio expressing the **efficiency** (sugar on sugar) of the factory based on the amount of the sugar produced (S_P) and sugar entered (S_E) the factory in sugarbeet (M_B = amount of beets and S_B = sugar content of beets). Sugar extraction (E) can be calculated by the following equation:

$$E = \frac{S_P}{S_E} \times 100 = \frac{S_P}{M_B S_B} \times 100 \tag{1.3}$$

Sugar recovery (R) is a percentage ratio expressing a rough estimate of the efficiency of the factory based on the purity of the thin juice and molasses. R can be calculated from the purity of the thin juice (P_J) and the purity of molasses (P_M) by a nonsugar elimination (NSE) formula (given in the Appendix):

$$R = \frac{P_J - P_M}{P_J(100 - P_M)} \times 10000 \tag{1.4}$$

NOTES

- **Extraction (*E*) is *mass dependent*, but recovery (*R*) is *purity dependent*. Unlike *E*, which represents the total sugar losses (losses to pulp, carbonation lime, and molasses), *R* represents only the sugar losses to molasses. Study the following example.**
- **The recovery formula, also known as the nonsugar-elimination (*NSE*) formula (see the Appendix), is used to express the efficiency of the stations of the factory involved with nonsugar elimination (mainly, the purification and crystallization stations).**

EXAMPLE 2

Given:

Beet processed	5050 t in 24 hours
Beet sucrose content (S_B)	16.55%
Thin juice purity (P_J)	91.2%
Molasses purity (P_M)	60.2%
Sugar produced (S_{PRO})	673.5 t

Calculate:

Extraction of the factory (*E*)

Recovery of the factory (*R*)

$$E = \frac{673.5}{5050 \times 16.55/100} \times 100 = 80.6\%$$

$$R = \frac{91.2 - 60.2}{91.2\,(100 - 60.2)} \times 10000 = 85.4\%$$

At normal operation and when normal beets are processed, approximately 80% of the sugar in the sugarbeet is extracted (removed) in the form of granulated-refined sugar. Higher extractions are expected from factories with the molasses desugaring by chromatographic (**MDC) process**.

The extraction of sugar is also expressed in kilograms (or pounds) of sugar per ton of beet processed. Usually, the sugar factory processing sugarbeet with about 17% sugar produces 120 to 150 kg sugar per ton of beet (240 to 300 lb sugar per short ton of beet). In the United States, the term **cwt/t** (reads 100 weights per ton of beet) is also used to indicate the sugar production based on 100 pounds of sugar per ton as beet procesed.

The **overall extraction** (yield at the field and at the factory level) is expressed as metric tons sugar per hectare (ha) or short tons sugar per acre (in the United States). Numerically, comparing overall extractions is not always suitable because the **stand count** (the number of sugarbeets per row) and the amount of rows per certain land are different from one region to another, making a difference in the amount of sugarbeet harvested per ha (in the metric system), or per acre (in the British system).

The **sugar losses** of a sugar factory are obviously linked to its extraction (yield) rate. For example, a factory with 80% extraction loses 20% of the sugar during operations. The main portion of the operational losses ends up in molasses (about 15%). There is some sugar loss in the **pulp** (exhausted beet cossettes) and **carbonation-lime residue** (CLR, limecake, spent lime left after juice purification). A few other sources of small losses, such as those during **beet fluming** (transport of beets by force of water), are known as **unaccounted losses** (unknown losses). The following table shows the yield of a typical conventional beet-sugar factory without and with the molasses desugaring by chromatographic (MDC) process.

	Without MDC	With MDC
Sugar extracted	82%	90%
Sugar lost to molasses	14%	6%
Sugar lost to beet pulp	2%	2%
Sugar lost to carbonation-lime residue	1%	1%
Unaccounted losses	1%	1%

NOTE

One of the surest ways to increase the efficiency of a beet-sugar factory is to decrease the losses of sucrose in the pulp, carbonation-lime residue, and particularly in molasses. Sugar left in molasses (even if it is to be desugared later) is valued at a much lower price than granulated-refined sugar. Therefore, it is important to reduce the sugar losses in molasses to the lowest possible level. (See section 17 of Chapter 3 and Chapter 8 for more information).

BY-PRODUCTS OF THE BEET-SUGAR FACTORY

In 100 kg sugarbeet, about 75 kg is water and 25 kg is dry substance (see Chapter 3 for more on sugarbeet composition). About 15 kg of the dry substance (*DS*) contained in 100 kg beet ends up in the form of a product (the amount of sugar produced is about one-seventh the weight of the beets), and the remaining 10 kg is a combination of by-products. This means that about 60% of the beet *DS* is turned into product and 40% into by-product (for a typical analysis of by-products, see Chapter 4).

The by-products of the beet-sugar factory are the following:

- Pulp
- Carbonation-lime residue (CLR)
- Molasses

Pulp

Pulp consists of desugared beet cossettes, which are usually dried and processed as animal feed. The dried pulp is often pelletized to increase bulk density to minimize shipping costs to distant markets. The dried pulp is valuable cattle feed because it supplies carbohydrates, proteins, and minerals.

Carbonation-Lime Residue

Sugar factories use lime, in the form of **calcium hydroxide** [$Ca(OH)_2$, known as milk of lime] in the juice-purification station, to improve the quality of the beet juice. After the lime has been used, the juice is mixed with **carbon dioxide gas** (CO_2), which precipitates lime again in the form of **calcium carbonate** ($CaCO_3$). The calcium carbonate is then concentrated using **cake filters** (rotary drum filters or filter presses) to produce **carbonation-lime** residue (CLR). The CLR stockpiled in the factory can take up space. Therefore, the sugar industry has been trying to find different uses for this material.

Carbonation-lime residue consists of about 80% $CaCO_3$ as dry substance. It is ideal for use as an agricultural soil-enhancer and pH adjuster because the lime also gives some of the nutritients separated from the sugarbeet juice back to the soil. Besides calcium, it contains other minerals such as Na and K that improve the soil. Carbonation lime has also proved valuable to sugarbeet growers. It is less expensive than other lime fertilizers.

Molasses

Molasses is the runoff syrup from the final stage of crystallization, at which stage further separation of the sugar is not possible with conventional equipment. Beet molasses must not contain less than 48% sugar (namely sucrose and invert sugar) and 79.5% *DS* when is offered to consumers. Molasses produced by beet-sugar factories usually contains about 50% sugar and 80% *DS*. It is the most valuable by-product of the sugar factory. Its market includes the yeast fermentation, pharmaceutical, and animal feed industries. It is desugared (mainly in the United States) using the molasses desugaring by chromatographic (**MDC**) process.

When a factory is equipped with the MDC process (see Chapter 8), it produces two more by-products:

- CMS
- Betaine

CMS (concentrated molasses solids) contains mainly minerals and low amounts of sucrose that is usually sold to the animal feed industry.

Betaine is one of the nitrogenous compounds present in sugarbeet that ends up in molasses. It makes up about 5% of the *DS* in molasses and is one of sugarbeet's most valuable components. In the molasses desugaring process, a considerable amount of high-purity betaine can be recovered and sold as a **liquid feed supplement**, primarily to the animal feed industry. For a typical analysis of CMS and betaine, refer to Chapter 4.

Extract (sucrose-rich fraction) is the product of the desugaring process processed in the sugar-end into sugar and **extract molasses** (secondary molasses). Typically, the production of such molasses is about 10 to 12% on the mass of molasses desugared or about 0.5 to 0.6% on beet (**OB**) processed if all the molasses from the beet crop is desugared.

MANAGERIAL STRUCTURE AND OWNERSHIP

A typical beet-sugar company consists of the following departments:

- Agriculture
- Operation

- Finance
- Marketing
- Purchasing
- Human resources

Engineering, process control, environmental, computer applications, research, communications, traffic, and safety are also sections of a sugar company. These may be independent departments or subsidiary departments.

A typical U.S. sugar factory with about 10000 t/day sugarbeet capacity has about 250 sugarbeet growers, producing about 1.5 Mt of beets per campaign in about 75000 acres (30000 ha) of land. The beet-processing campaign lasts about 150 days. The number of employees of such a factory is as follows:

Year-round employees	100
Seasonal employees	120
Harvest employees	60
Salaried employees	40

The **ownership** of sugar companies worldwide is of four types:

- **Privately owned companies:** Owned by one or a few individuals (usually members of same family)
- **Shareholder-owned companies:** Owned by holders of shares in company earnings
- **Government-owned companies:** Owned by the government
- **Cooperative companies:** Owned by sugarbeet farmers

Cooperative sugar companies (simply, co-ops) were established in the United States to manage the factory facilities more efficiently. These companies are owned by the sugar growers (see the Economy of Sugar subsection in Section 1 of this chapter for more information on such co-ops).

CHAPTER 1

SECTION 3

SUGAR TERMINOLOGY

SUBSECTIONS

- Sugar Terms Used in This Book
- Aqueous Solutions
- Juice, Syrup, and Liquor
- Pure Sucrose Solutions
- Impure Sucrose Solutions
- Dry-substance Content
- Sucrose Content
- Nonsucrose Content
- Purity
- Hardness
- Alkalinity
- Measured as CaO
- Coloring Substances
- Percentages Used in Sugar Technology

ABOUT THIS SECTION **This section** familiarizes you with special sugar terms. The **sugar terminology** covered in this section includes the special names and phrases used in the sugar industry for oral and written communication. Brix, purity, nonsugars (nonsucroses), impure sucrose solution, and affination are examples of industry terms.

Every day, people involved with the sugar industry encounter many special sugar terms. Although they might know the general meaning of these terms, some may not be fully aware of their precise meanings. Therefore, it is important to learn the definitions of sugar terms, which is the main goal of this section. In addition, this section gives the proper nomenclature used in our industry.

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The sugar industry is old. Its origin goes back to a prescientific time, so sugar terminology is due more to tradition than to application of **technically correct terms**. Sugar technology would be easier to understand if our industry had **standard sugar terminology** (SST). Some terms are in use in only a single country or even in a single factory within a country with several factories. This creates difficulties for sugar technologists discussing sugar processes. Because there is no SST, the sugar literature can be confusing as well. Sugar technologists have tried, in the meeting of the International Commission for Sugar Technology (known by its French Acronym, CITS) in 1975, to standardize the special sugar terms, but thus far, the result has not been satisfactory. Today, considerable confusion in the definition of some sugar terms still exists in the sugar literature.

Once a comprehensive SST is established, there will be one correct term for every concept. Until then, it is every sugar technologist's responsibility to try to use the most exact and unified system of names possible.

SUGAR TERMS USED IN THIS BOOK

The use of **exact** (precise and accurate) terms affects the clarity of our messages and learning ability. Unfortunately, the sugar literature contains some inexact and ambiguous terms as well as some **multiple terms** (different terms to express one particular concept), leading to confusion and error.

Sugar technologists, even those in the same country, often use different terms to indicate the same concept. This practice can be seen in some sugar technology books in which different chapters were written by different authors.

The following are a few examples of multiple-term usage. Let us start with sugar. Today, many different names are used to refer to sugar that is in granulated-refined form:

- White sugar
- Crystal sugar
- Refined sugar
- Granulated sugar
- Crystalline sugar
- Crystallized sugar
- Refined white sugar
- Granulated white sugar
- White granulated sugar
- White centrifugal sugar
- Crystallized white sugar

Sugar is one of the most widely traded products in the international market. Individuals unfamiliar with the sugar industry might think that each of the above-given terms indicates a specific type of sugar. Foremost in importance is the need for a standard name to be used for sugar to distinguish among the types of sugars.

North American sugar technologists use *granulated sugar* because sugar consists of *granules* (crystals). Some European technologists use *refined sugar*, *white sugar*, or *refined-white sugar* because sugar is refined and white as opposed to raw sugar, which is unrefined and off-white.

Most sugar technologists correctly combine two terms to distinguish sugar from raw and powdered sugars. The term *refined-white sugar* differentiates sugar from raw sugar (raw sugar is neither refined nor white) but does not exclude powdered sugar because both sugar and powdered sugar are refined and white. In general, it seems that two adjectives can describe sugar better. The question is which combination is the most suitable.

To eliminate ambiguity, the term **granulated-refined (GR) sugar** is used in this book for two simple reasons. First, it clearly distinguishes the type of sugar from both raw and powdered sugars. Second, it is familiar to sugar technologists because North American sugar technologists use the **granulated** prefix while most Europeans use **refined**. The suggested name is therefore a combination of both. **Sugar** and **sucrose** are used interchangeably, as is usual in sugar literature.

Another example of the confusion created by the nonstandardization of terms is that many of us (sugar technologists and chemists) still use the term **Brix** (after A. Brix, the inventor of the hydrometer scale) as if it were a real substance, although it is used as a unit of refractometer scales (degrees). In the sugar industry, Brix is used as the percentage (by mass) of **dry substance** (*DS*) of sugar solutions, measured by a refractometer. Some even use the term Brix in calculations as a measure of the mass of a substance: for example, so many tons Brix in so many tons of molasses. Some sugar technologists use instead the term **percent refractometric dry substance** (% *RDS*), which is accurate and self-defining. In this book, the terms *RDS* or *DS* are used interchangeably.

The same case applies to the term **pol** (short for polarization), which is the percentage of sucrose (by mass) measured by a polarimeter. Pol is degree of rotation of the polarized light in a solution, but the term is used as if it were a real substance. The more precise term that is self-defining is **polarimetric sucrose** (polarimetric sugar), which will be used in this book.

Some terms were chosen for this book because they invoke their antonyms (opposite forms). For example, instead of using *technical sucrose solution*, I chose **impure sucrose solution** because it is an antonym for **pure sucrose solution**. In addition, the terms *purity* and *impurity* are common in our industry.

The terms used to describe **in-process products** (juices, syrups, liquors, and massecuites) are mostly distinguished by the same prefixes as the process from which they originated. For example, from the two common terms *diffusion juice* and *raw juice*, the term **diffusion juice** is chosen because the juice is the product of the diffusion process.

Multiple-term usage gets more confusing when we read about the crystallization process. Sometimes it is difficult for an experienced person to follow a text because different names are used to denote the same syrup. For example, the following names are commonly used to denote the mother liquor from the first stage of crystallization after the massecuite is centrifuged:

- A-syrup
- A-green syrup
- High-green syrup
- Green runoff syrup
- Intermediate syrup

In this book, the term **high-green syrup** or **A-green syrup** is used. In addition, nondescriptive crystallization terms, such as green sugar (the sugar that has not been properly centrifuged), hardening the grain (speeding the growth of formed crystals), bringing pan together, and after products are not used. Furthermore, instead of Latin crystallization words, their equivalent English words are used (e.g., stable zone is used instead of labile zone).

Following are some of the sugar terms used in this book:

- **Granulated-refined (GR) sugar** instead of all other names for table sugar (sugar and sucrose are also used interchangeably)
- **Beet-end** for beet washing through juice evaporation instead of raw side or raw end
- **Sugar-end** for the crystallization section of the factory instead of boiling house, sugar house, or recovery house
- **Refractometric dry substance** (*RDS*) or simply dry substance (*DS*) instead of Brix
- **Polarimetric sugar** (*PS*) instead of pol
- **Polarimeter** instead of polariscope or saccharimeter
- **Purity** (*P*) instead of coefficient of purity (*q*)
- **Impure sucrose solution** instead of technical sucrose solution
- **Diffusion juice** (the product of the diffusion process) instead of raw juice
- **Carbonation gas** for gas produced in the limekiln containing about 33% CO_2, 63% N_2, and 3% O_2)
- **Carbonation-lime residue** (CLR) or limecake for carbonation mud after its concentration with **cake filters** (filter presses or rotary-drum filter)

Now that we have learned about some of the shortcomings in sugar terminology, let us define some sugar terms.

AQUEOUS SOLUTIONS

Aqueous solutions are the solutions in which *water* is the **solvent** (dissolving substance). Water's ability to dissolve many substances is considered its most valuable property. Even some alcohols, such as ethanol (C_2H_5OH) and ethylene glycol (used in cars in the antifreeze-coolant solution), are soluble in water.

Sucrose solution (sugar dissolved in water), salt solution (salt dissolved in water), carbonated water (carbon dioxide dissolved in water), and alcohol solution (ethanol dissolved in water) are examples of aqueous solutions. In these examples, sugar, salt, carbon dioxide, and ethanol are the solutes, and water is the solvent.

In general, the sugar found in a sugar factory can take three forms:

- **Crystalline sugar:** A solid containing repeating organized molecules, such as raw sugar
- **Sugar-aqueous solution:** A solution containing sugar and nonsugars, such as diffusion juice
- **Amorphous sugar:** A solid containing broken, unorganized, and shapeless molecules of sugar, such as powdered sugar

In sugar technology, we always deal with aqueous solutions in which sugar and nonsugars are the **solutes** (the dissolved substance) of the solutions, and water is the solvent. (Note: Sugar

factory juices, syrups, and liquors belong to aqueous solutions because the dissolving media is water.)

Following are three important properties of aqueous solutions:

- When an aqueous solution evaporates, only the solvent evaporates, not the solutes. For example, in thin-juice evaporation, only water evaporates.
- Aqueous solutions, in which sugar is dissolved, boil at a higher temperature (*the higher the concentration of a sugar solution, the higher is its boiling point*) than water alone.
- Aqueous solutions, in which sugar is dissolved, freeze at a lower temperature (*the higher the concentration of a sugar solution, the lower is its freezing point*) than water alone.

NOTE

The aq (abbreviation for aqueous) in parentheses after the formula of an acid or base shows that the acid or base is diluted in water to lower concentration: for example, H_2SO_4 (*aq*).

JUICE, SYRUP, AND LIQUOR

No sharp boundary divides impure sugar solutions (juice, syrup, and liquor). However, the following explanations work to define them:

Juice is an impure sugar solution with a dry substance (*DS*) content up to 55%. Diffusion juice (the product of the diffusion process with about 15% *DS*) and thick juice (the product of the evaporation process with about 55% *DS*) are examples of juices.

Syrup is the mother liquor (runoff syrup) of a **massecuite** that is separated from sugar crystals by the centrifuging process and nearly saturated with sugar. High-green (A-green) syrup (runoff from centrifuging white massecuite) and low-green (B-green) syrup are examples of syrup.

Liquor is thick juice (the product of the evaporation station) concentrated to a definite dry-substance content (by melting some raw sugar in it), filtered, sometimes even decolorized, and used as feed in the crystallization process. Standard liquor (the feed to the first-crystallization stage) is the only liquor of the beet-sugar factory.

Molasses does not belong to any of the preceding classes because it is a by-product, not an in-process product.

NOTE

In-process products (or simply in-products) is a term used in this book to denote juices, syrups, liquors, and massecuites because these are the products of individual stations in a sugar factory as opposed to the final product of the factory, which is sugar. The by-products are pulp, carbonation lime, and molasses. Because of the impurities in the in-process products, they do not meet the *pure-sucrose solutions* standard.

PURE SUCROSE SOLUTIONS

Sugar technology classifies sucrose solutions as pure and impure. A **pure sucrose** (PS) **solution** is a solution of sucrose dissolved in water. Because **PS-solutions** do not contain any nonsugars (*NS*), the purity (*P*) of these solutions is 100%:

$$P = 100 - NS$$
$$P = 100 - 0 = 100\% \tag{1.5}$$

Pure sucrose solutions have different properties than pure water. The following are a few properties of pure sucrose solutions compared with pure water:

- **Density:** The density of a PS-solution at any concentration is higher than water (Figure 1.7).

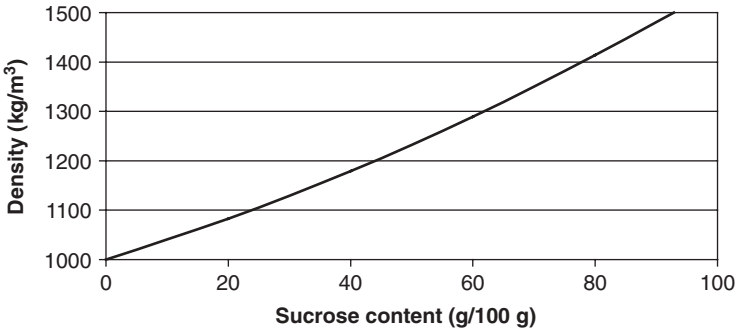


Figure 1.7 Density of pure sucrose solutions

- **Solubility:** The solubility of sucrose in water depends only on the temperature of the solution and increases considerably with increasing temperature.
- **Freezing point** (*FP*): The boiling point of PS-**solutions** is lower than pure water because the *FP* is proportional to the concentration of the solution (*the higher the concentration of a solution, the lower is its FP*).

NOTE

The properties of pure sucrose solutions are important in sugar technology because they are used as the standard for comparison with *impure sucrose solutions.*

IMPURE SUCROSE SOLUTIONS

An **impure sucrose** (IS) **solution** (technical sucrose solution) is a solution of sugar and nonsugars with different dry substance concentrations. Because **IS-solutions** contain some nonsugars, their purities are less than 100% (usually between 35 and 95%). *All in-process products and molasses belong to the impure sucrose solutions group.* Molasses with about 60% purity, diffusion juice with about 88% purity, and standard liquor with about 93% purity are examples of impure sucrose solutions.

Following are the effect of the purities on the properties of impure sucrose solutions:

- **Color:** The color of IS-solutions darkens as the purity decreases (impurities increase).
- **Solubility of sucrose:** Often the solubility of IS-solutions increases as the purity decreases, as shown in Figure 1.8. The solubility of sucrose in the presence of various nonsugars is dif-

ferent than in pure water. For example, chemicals such as CaO, $CaCO_3$, $MgCO_3$, and $CaSO_4$ can be dissolved in sucrose-containing solutions but not easily in pure water.

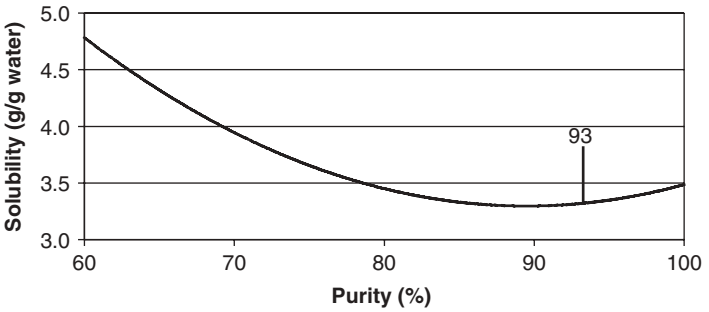


Figure 1.8 Effect of purity on solubility at 75°C

- **Viscosity:** The viscosity of IS-solutions increases as the purity increases, (see Figure 1.9).

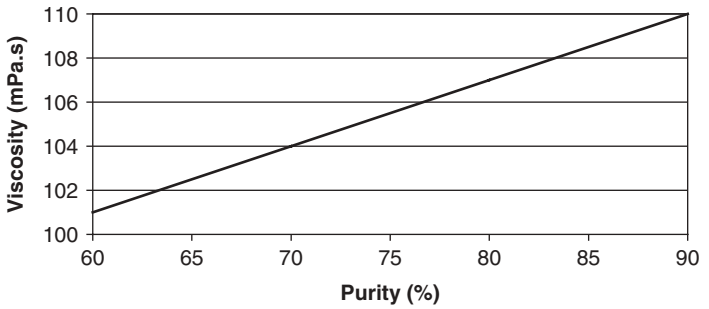


Figure 1.9 Effect of purity on viscosity at 80% *DS* and 75°C

- **Boiling-point elevation** (*BPE*): The boiling-point elevation of IS-solutions increases as the purity decreases, as shown in Figure 1.10.

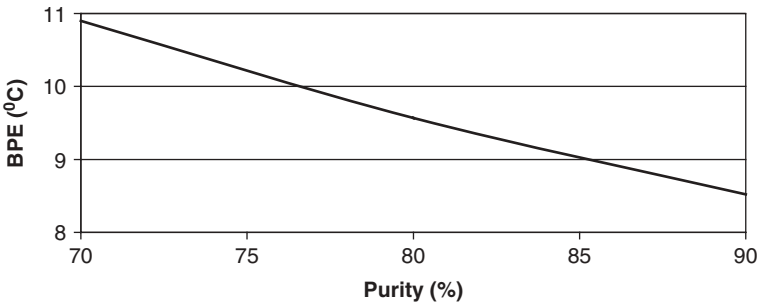


Figure 1.10 Effect of purity on *BPE* at 80% *DS* and 70°C

- **Specific conductivity:** The specific conductivity (k) of IS-solutions increases as the purity decreases, as shown in Figure 1.11.

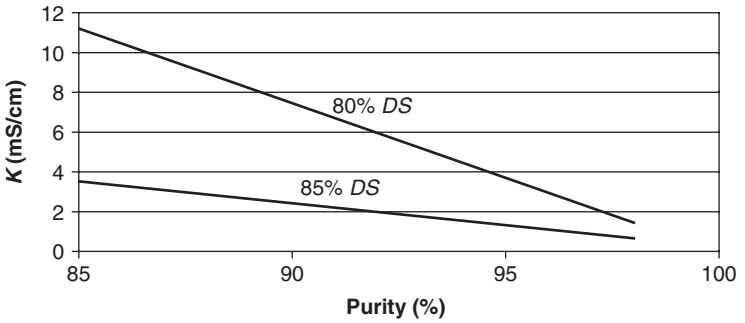


Figure 1.11 Effect of purity on conductivity (*k*)

- **Density** (*d*): The density of IS-solutions increases as the purity decreases.
- **Refractive index** (*RI*): *RI* of IS-solutions increases as the purity decreases.
- **Specific-heat capacity** (C_P): C_P of IS-solutions decreases as the purity decreases.
- **Surface tension:** The surface tension of IS-solutions increases as the purity decreases.
- **Solubility of lime:** The solubility of lime (CaO) in IS-solutions increases as the purity decreases. (The solubility of lime in IS-solutions is greater than in pure sucrose solution at the same *DS*.)

NOTES

- **The goal of sugar technology is to remove the impurities from sucrose solutions and produce sugar that consists of pure crystals. Therefore, sugar technology mainly concentrates on improving impure sucrose solutions.**
- **The properties of pure sucrose solutions are easier to investigate than impure solutions because each nonsugar behaves differently.**
- **The properties of impure sucrose solutions with a *DS* below 60% and purity above 90% are similar to those of pure sucrose solutions at the same *DS* and temperature. The values of pure sucrose solutions within this range are applicable to the impure sucrose solutions with sufficient accuracy. Therefore, in sugar processing, most tables are based on the properties of the pure sucrose solutions.**
- **Most laboratory instruments, such as the refractometer and the polarimeter, are therefore calibrated to the pure sucrose solutions.**
- **The properties of impure sucrose solutions and their behavior at each step of sugar processing, particularly in the purification and crystallization stations, are the most studied by sugar technologists. In fact, the natures of these two types of solutions are the dominant interest of sugar technology.**

DRY-SUBSTANCE CONTENT

Dry substance (*DS*) is the solid content of a solution given as a mass percentage (% m/m). *DS* represents the mass of the solution that remains after all water has been evaporated. Therefore, the percentage of *DS* of a juice represents the total sucrose and nonsucroses in 100 g of that juice. The *DS* content can be found by subtracting the water content (*W*) from 100:

$$DS = 100 - W\ \%\ \text{by mass} \tag{1.6}$$

A 60% *DS* solution represents a solution that contains 40 g water. In other words, 100 g of this solution contains 60 g *DS*. Two methods are used to determine the dry substance content of sugar solutions:

- Oven-drying method
- Refractometric method

In the drying method, *DS* is measured before and after the sample has oven dried at 105°C for two to three hours or to a constant mass. However, in sugar laboratories the refractometric method is used for almost all samples for the following two reasons:

- The results measured by the refractometric method and by the drying method are very close.
- Determination is faster by the refractometric method (about 3 minutes) compared to the oven-drying method (about 3 hours).

The dry substance content measured by refractometer expressed as mass percentage is called the **refractometric dry substance** (*RDS*). A **refractometer** is an instrument that measures the **refractive index** (*RI*) of a solution and converts it to % *DS*. The result measured by the refractometer is also called Brix or **degrees Brix** (°Brix) and has the same meaning as % *RDS*.

NOTES

- **In most sugar literature, as well as in this book, the term dry substance (*DS*) means that the sample was measured by the refractometric method, so *DS* and *RDS* have the same meaning.**
- **In pure sucrose solution, the *RDS* represents the sucrose content because sucrose is the only dry substance of the solution. But, if the solution is an impure sucrose solution, the *RDS* does not represent the sucrose content because the refractive index (*RI*) of sucrose and nonsucrose substances differs. In other words, a 1% nonsucrose solution does not change the *RI* as a 1% sucrose solution does (*the RI of impure sucrose solutions increases as the purity decreases*).**
- **For more information about *RDS* and methods of measuring it, refer to the Refractometer subsection in Section 2 of Chapter 11.**

SUCROSE CONTENT

Sucrose content is the amount of **sucrose** (sugar) in a solution identified by mass percentage (% m/m). In pure sucrose solutions, the mass of sucrose can be found by deducting the mass of water from 100. A 20% sucrose solution, for example, represents a solution that has 20 g sucrose and 80 g water. In impure sucrose solutions, the mass of sucrose (*S*) equals the total mass of water (*W*) and nonsucroses (*NS*) subtracted from 100.

$$S = 100 - (W + NS)\ \ \%\ \text{by mass} \tag{1.7}$$

Like *DS*, the sucrose content in sugar laboratories is measured by two methods:

- Inversion method
- Polarimetric method

However, the polarimetric method is usually used because

- The results measured by the polarimetric method and by the inversion method are very close.
- Determination is fast by the polarimetric method (about 3 minutes), and slow by the inversion method (about 3 hours).

The sucrose content measured by a polarimeter is called **polarimetric sucrose** (*PS*). The **polarimeter** is an instrument that measures the degrees of **rotation** (bending) of polarized light in a solution containing optically active substances (e.g., sucrose) and then converts the degrees into percentage of sucrose. The result measured by polarimeter is called direct polarization, or **pol**, and has the same meaning as % polarimetric sucrose. (For more information about polarimetry and sucrose determination, refer to the Polarimeter subsection in Section 2 of Chapter 11.)

NONSUCROSE CONTENT

Nonsucrose substances (*NS*, simply **nonsucroses** or **nonsugars**) refer to impurities in beet-juice or in in-process products. Nonsucroses include all dissolved substances except sucrose. The nonsucrose content is equal to the dry substances (*DS*) content minus sucrose (*S*) content.

$$NS = DS - S \tag{1.8}$$

Glucose, fructose, raffinose, amino acids, coloring substances, and different minerals are examples of nonsugars. In sugar processing, the nonsugars (impurities) are unwanted, as they have to be separated from sucrose (wanted) in several steps. Nonsugars (*NS*) are divided into the following groups:

- Nitrogenous *NS*: For example, amino acids and betaine
- Non-nitrogenous *NS*: For example, glucose, fructose, and organic acids
- Inorganic *NS*: For example, calcium, magnesium, potassium, and sodium
- Coloring *NS* (colorants): For example, melanins

NOTES

- **The efficiency of the sugar factory is dependent not only on the sucrose content of the sugarbeet but also on the nonsucrose content. The content, nature, and behavior of nonsucrose substances during beet processing, and the methods of separating them from sucrose, are the most-talked-about subjects in sugar technology. In fact, if nonsugars did not exist in sugarbeet, sugar technology would be a much less time-consuming subject to learn than it is now.**
- **Pulp (sugar-exhausted sugarbeet) and other substances of sugarbeet that are left with the pulp do not belong to nonsugars.**
- **The difference in type and amount of *NS* present in sugarbeet and sugarcane is the main reason why the production of sugar from these two crops differs.**

PURITY

Purity (*P*) is a sugar term used to describe the percentage (by mass) of sucrose in the total dry substance (*DS*) of an in-process product (juices, syrups, and liquors). Purity is the most common

term used by sugar technologists. For example, a juice with a purity of 90% means that 100 g *DS* of this juice (after all its water is evaporated) contains 90 g of sucrose and 10 g of nonsucrose substances. Consumer-quality sugar has a purity of almost 100% because it contains almost no nonsugar (impurities).

Purity is used to compare the quality of in-process products and, consequently, the efficiency of individual stations of the factory. Higher purity in one product as compared to another is an indication of higher quality (higher sucrose and lower nonsucrose content). In the case of molasses, the sugar technologists try to produce molasses with the lowest possible purity. This is because the sugar lost in molasses is considered sugar loss (*the lower the purity of molasses, the lower is the amount of sugar lost to molasses and, consequently, the higher is the sugar production*). For more information on the purity of molasses, refer to Section 17 of Chapter 3. Mathematically, purity (*P*) is the product of the percentage of sugar (*S*) divided by the percentage of *DS*, multiplied by 100.

$$P = \frac{S}{DS} \times 100 \tag{1.9}$$

The nonsucrose (*NS*) content is given as

$$NS = DS - S \tag{1.10}$$

To specify purity, we must indicate the method of sucrose content determination. When the sucrose content is determined by the polarimetric method and *DS* by the refractometric method (*RDS*), the result is called **apparent** (approximate) **purity**. When the sucrose content is determined by the inversion method (true sucrose) and the dry substance is determined by the refractometric method, the result is called **true purity**.

$$\%\ \text{Apparent purity} = \frac{\text{Polarimetric sucrose}}{RDS} \times 100 \tag{1.11}$$

$$\%\ \text{True purity} = \frac{\text{True sucrose}}{RDS} \times 100 \tag{1.12}$$

EXAMPLE 3

Given:

Dry substance (*DS*) of thick juice	62.0%
Sucrose content (*S*) of thick juice	57.1%

Calculate:

Purity of thick juice (*P*)
Water content (*W*)
Nonsucrose (*NS*) content

$$P = \frac{S}{DS} \times 100 = \frac{57.1}{62.0} \times 100 = 92.1\%$$

$$W = 100 - DS = 100 - 62.0 = 38.0\%$$

$$NS = DS - S = 62.0 - 57.1 = 4.9\%$$

NOTES

- **The equation for determining purity was derived by K. Balling (a Czech sugar scientist from Prague and the inventor of the Balling hydrometer). This equation probably has served the industry better than any other invention. Let us see how the purity formula has simplified sugar operations. Assume that the purity of the diffusion juice is initially 88% but increases to 90% during purification. This simply means that the quality of the juice was improved by 2%. Imagine the difficulty of comparing these two products without using the purity equation because the products have to be compared by two values: dry substance and sugar contents.**
- ***Purity does not change by concentration or dilution*** **because purity is not relevant to water content. This is because during the evaporation or dilution process, water is the only substance whose content changes. In other words, S to DS ratio does not change by decreasing (in the case of evaporation) or increasing (in the case of dilution) the amount of water. For example, the purity of the thin juice does not change during the evaporation process, assuming that no *decomposition of sucrose occurs*. Analyzing thick juice for purity is of no significance in the evaporation operation. The lab does not usually measure purity of thick juice because its value is the same as with thin juice, which is tested on a regular bases.**
- **In chemistry, a pure material is defined as one consisting of a single element or a single compound. For example, pure sugar consists of virtually nothing but *sucrose*. On the other hand, impure material contains two or more elements or compounds. It is *almost impossible to produce an absolute pure substance* that truly consists of 100% of a single material because atoms are very small. If, for example, one atom out of million were different, 100% purity would not be achieved. Sucrose, in its granulated-refined form, is considered the world's purest food product, with a purity of 99.95%. The remaining 0.05% is water (0.03%) and a few other elements in trace amounts (mostly inorganic ashes).**

HARDNESS

Hardness (limesalts) is the amount of soluble salts of calcium (Ca) and magnesium (Mg) in a solution. During beet juice evaporation, some hardness precipitates in the form of calcium carbonate ($CaCO_3$) and calcium oxalate (CaC_2O_4), producing rocklike **scale** in the evaporator's tubes. This happens because during evaporation, concentration is increased and solubility of limesalts is decreased. In addition to scaling problem in evaporators, high hardness-content juice creates difficulties in other steps of process (particularly in crystallization). Therefore, testing different in-process products for hardness content is one of the main jobs of the laboratory.

In the sugar industry, the hardness content is expressed as mg/100 *DS* (read milligrams in 100 grams of dry substance) of the sample. For example, a juice with 120 mg hardness means that 100 g *DS* of this juice contains 120 mg of hardness, measured as CaO. (The "measured as CaO" is described later.) This is equal to 1200 ppm (1200 mg hardness per one million mg *DS*). Assuming that the *DS* of the sample is 15%, this equates to $120 \times 15\% = 18$ mg in 100 g of the sample, which is called **% on sample**.

The hardness substances are formed mainly during the first-carbonation process. Under normal operation conditions, the hardness present in **thin juice** (the product of the purification station) depends on the sugarbeets' nature and origin. The contents of 50 to 200 mg are considered normal, but the hardness can get quite high (up to 600) when a factory processes **damaged sugarbeet** because of either a long beet storage period or frost. The hardness content of thin juice in factories is considered normal in the western part of the United States and many parts of Europe but is particularly high in some geographical areas where soils are highly alkaline. This is the case, for example, in factories in Michigan (in the United States), Morocco,

Spain, and Iran, where the thin juice usually contains high amounts of hardness (150 to 600) and low natural alkalinity.

In general, hardness is divided in two groups: soluble and insoluble. In sugar technology, the term hardness refers to the **soluble** (dissolved) group. The **soluble hardness** (further just hardness or limesalts) is not filterable and therefore stays with the juice throughout processing. As mentioned, some hardness precipitates during evaporation and some passes the evaporation and crystallization process and ends up in molasses. High hardness causes the following operational problems:

- Decreases crystallization efficiency
- Decreases molecule movement
- Decreases sucrose recovery
- Increases juice viscosity

NOTE

- **Precipitation of hardness in evaporator tubes is important because of evaporator *scaling*, which requires cleaning. In sugar terms, the cleaning process is known as evaporator boil-out. In terms of evaporator scaling, some hardness substances are harmful (harmful hardness) and some are *less harmful*. For example, hardness in thin juice (produced in one sugarbeet growing area) in the amount of 300 mg/100 *DS* can create a need for frequent evaporator cleaning, while the same amount in the other areas do not necessitate as many boil-outs. This depends on the *type of hardness* in the juice. For example, the hardness in beets grown in Michigan belongs to the less harmful group, although the quantity is high (200 to 600 is not unusual).**

ALKALINITY

Alkalinity is the total amount of carbonate (CO_3^{2-}), bicarbonate (HCO_3^-), and hydroxide (OH^-) of Ca, Mg, Na, and K in a solution. In beet operations, alkalinity has been found to be a good indication of first and second carbonation-juice quality, so alkalinity is an important analysis in every sugar laboratory. In sugar technology, alkalinity is divided into two groups:

- **Natural alkalinity**: Amount of dissolved K and Na *carbonates* present in the juice.
- **Effective alkalinity**: Amount of dissolved K and Na *carbonates and bicarbonates* present in the juice. Effective alkalinity is a good indicator of juice behavior during further stations of operations (particularly in the evaporation station).

Buffering capacity refers to the effective alkalinity of the purified juice. The juice with low effective alkalinity is called **low-buffered juice**. When low-buffered juice enters the evaporators, its **pH** decreases and its color increases, but it does not scale the evaporators considerably.

NOTES

- **For a complete explanation about different types of alkalinity, see Section 10 of Chapter 3.**
- **The determination of the alkalinity of first- and second-carbonation juice are important routine tests, and lab analysts provide them on an hourly basis and express the results in gram or mg/100 mL of sample measured as CaO.**

MEASURED AS CaO

Measured as CaO (or simply as CaO) or **measured as $CaCO_3$** (or as $CaCO_3$) are terms often used in sugar technology to express the alkalinity and hardness (limesalts) content of in-process products. As stated, hardness consists of Ca and Mg salts in the juice. If the calcium salts were reported as Ca and magnesium salts as Mg, that way of expression would create difficulties because they cannot be added together.

Likewise in the case of alkalinity, reporting carbonate as $CaCO_3$, bicarbonate as $Ca(HCO_3)_2$, and hydroxide as $Ca(OH)_2$ would create difficulties. To simplify the expression, hardness and alkalinity are reported as CaO.

Expressing "as CaO" or "as $CaCO_3$" essentially establishes an equal denominator for all components, resulting in a direct relationship of the different components present in the sample. In other words, converting salts as Ca, as Mg, as HCO_3, and so on, to the common term as CaO is convenient because 1 g of calcium, for example, can be considered as 1 g of magnesium because they have the same denominator (the equivalent of CaO).

NOTES

- **Expressing as CaO is much like converting European dollars into American dollars, since the value of the money stays the same (at the time of conversion).**
- **For ways to calculate substances "as CaO" refer to Chapter 6.**
- **In the laboratory, chemists prepare a standard acid solution (an acid solution with exact concentration) based on the ratio of equivalence of acid to equivalence of CaO. Then, the hardness content of the sample is determined from titration of a measured volume of the sample against the standard acid. The result indicates the total hardness of the sample as CaO.**
- **Alkalinity, pH, and hardness content are important criteria of in-process products in a sugar factory. These are related characteristics. For example, a drop in pH during evaporation in the range of 0.2 to 0.4 is normal; anything above this is an indication of low effective alkalinity and high hardness content that, among other consequences, produces a juice with high coloring substances.**

COLORING SUBSTANCES

Coloring substances (colorants) are high-molecular compounds formed during the individual steps of sugarbeet processing, particularly during evaporation and crystallization. Most colorants, formed during processing, are the products of the **Maillard reaction**, which occurs at high temperatures between the hydroxyl group of reducing sugars and the amino groups of amino acids. (See Section 14 of Chapter 3 for more information about Maillard reaction.)

NOTES

- **The color of in-process products is important because the production of low-color sugar is desirable.**
- **In the sugar industry, the color of all in-process products, the final product (sugar), and molasses is measured by a spectrophotometer. The result is expressed in numbers and is independent of the observer's judgment. The unit for color expression is the ICUMSA unit (IU), which is also called ICUMSA-420 color. Thus unit is used internationally in the sugar industry. The ICUMSA (International Commission for Uniform Methods of Sugar Analysis) is an international organization that sets standards for the uniformity of analytical procedures in the sugar industry.**

PERCENTAGES USED IN SUGAR TECHNOLOGY

The **percentages** used in sugar technology are as follow:

- **Percent on dry substance** (% on *DS*): For example, thin juice with 125 mg CaO/100 *DS* (read milligrams CaO per 100 g *DS*) contains 125 mg CaO in 100 g of its dry substance. In conversation, this often simplifies as CaO on *DS*, or CaO on dry basis.
- **Percent on juice** (% on juice): For example, an alkalinity of first-carb juice of 0.09% on juice means there is 0.09 g CaO in 100 mL of juice. The amount of the **suspended solids** of the diffusion juice equal to 0.5% on juice means that 100 mL of juice contains 0.5 g suspended solids.
- **Percent on beet** (% OB): Usually the beet-sugar factory expresses most of the data in percentage of the processed beet mass and calls it **% on beet** (% OB). For example, a sugar loss in pulp of 0.2% OB means 0.2 ton (t) of sucrose is lost when 100 t of beets are processed.
- **Percent on sugar** (% OS): Some data in beet-sugar operations are related to either the amount of sugar in the beet or the amount of sugar produced. For example, a sugar loss in pulp of 1.5% OS means 1.5 t of sugar is lost per 100 t of sugar in beet. A steam consumption of 2.5% OS means that the factory consumes 2.5 t of steam to produce 100 t of sugar.

NOTE

Mass and weight are common terms that are incorrectly used interchangeably in technical literatures. The term mass is used in this book to the extent that it does not create any confusion. For example, in expressing percentages, percent by mass (% m/m) is used instead of percent by weight (% w/w), and molecular mass is used rather than molecular weight.

CHAPTER 1

SECTION 4

SUCROSE PROPERTIES

SUBSECTIONS

- Sucrose Configuration and Nomenclature
- Sucrose Formation
- Sucrose Density
- Sucrose Melting Point
- Sucrose Solutions' Boiling-Point Elevation
- Sucrose Solutions' Freezing-Point Depression
- Sucrose Solubility
- Sucrose Is Nonreducing
- Sucrose Is Nonionic
- Sucrose Acidic Reactions
- Sucrose Alkaline Reactions
- Sucrose Half-Life
- Sucrose Specific Heat
- Sucrose Specific Enthalpy
- Sucrochemistry

ABOUT THIS SECTION **This section** focuses primarily on the configuration and physical and chemical properties of sucrose (sugar). For example, you will learn that increasing the concentration of a solution by adding sucrose causes the boiling-point value to go up and the freezing-point value to go down.

The **sucrose molecule** ($C_{12}H_{22}O_{11}$) consists of 12 carbon atoms (C), 22 hydrogen (H), and 11 oxygen atoms (O). In percentages, the molecule contains 51.5% oxygen, 42.0% carbon, and 6.5% hydrogen. The molecular mass (weight) of sucrose is 342.3 g.

Beet-Sugar Handbook, by Mosen Asadi
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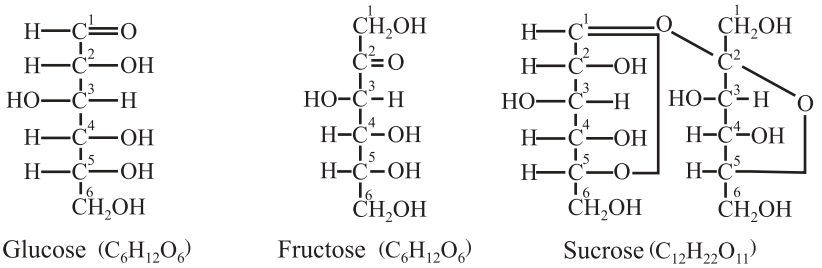


Figure 1.12 Structures and main sources of three simple sugars

Saccharides (from the Latin *saccharum* meaning sugar) is the scientific name for **sugars**. Sugars are in the **carbohydrates group** (sugars, starches, and dietary fibers). Glucose, fructose, and sucrose are the most known saccharides (see Figure 1.12).

SUCROSE CONFIGURATION AND NOMENCLATURE

Sucrose is a disaccharide, made from glucose and fructose rings, each with six carbon atoms. The Glucose molecule is a six-member ring compound, and fructose has five members. The rings in the sucrose molecule are connected (bonded) by a **glycosidic bond** (oxygen bond) in a covalent manner (see Atom and Molecule subsection, in Section 1 of Chapter 12 for definition of **covalent bond**). The glycosidic bond connects the functional group of the glucose molecule (an aldehyde group) to the functional group of the fructose molecule (a ketone group). (Note: A **functional group** (see also Glossary) defines a chemical family, and consists of a group of atoms in a molecule, which behaves as a unit when the molecule reacts with the molecule of other compound. For example, all 8 hydroxyl groups (OH) of sucrose can be replaced by methyl group (CH_3) of other compounds to form new organic compounds.

In the chemistry of sugars, the configuration of sugars is depicted in two ways:

- By the straight (Fischer) method
- By the cyclic (Haworth) method

Figure 1.13 shows the glucose, fructose, and sucrose molecules, drawn using both methods. In the **straight** (Fischer) method, the formula is written as a straight chain with the aldehyde (or ketone) group at the top (carbon-1 is at the top). The H and OH groups are written at the right and left of each **interior** (asymmetric) carbon atom. For example, in glucose and fructose, C–2 through C–5 are interior carbon atoms (see Figure 1.13). In the **cyclic** (Haworth) method, any

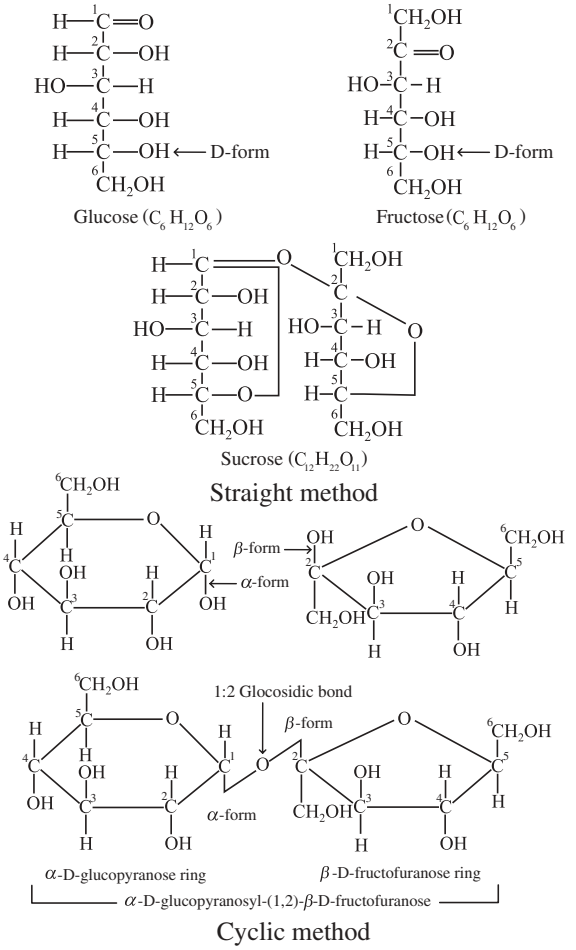


Figure 1.13 Glucose, fructose, and sucrose configuration, shown in the straight and cyclic methods

H and OH written to the right of the interior carbons in the straight method are placed below the ring, and any H or OH written to the left are placed above the ring.

In sugar **nomenclature** (naming rules), the name of sucrose is α-D-(+) glycopyranosyl-(1,2)-β-D-(−) fructofuranose because it consists of an α-D-(+) glycopyranose unit (ring) and a β-D-(−) fructofuranose ring joined by a glycosidic bond from carbon 1 on glucose to carbon 2 on fructose (1,2 glycosidic bond). The following six general rules related to the terminology of sugars (saccharides) clarify the naming system of any sugar as well as sucrose:

- The **pyranose** and **furanose** notations in the name of a sugar indicate its ring (rings) type. The six-member-ring sugars are known as pyranose (after the compound pyran). Five-member-ring sugars are called furanose (after the compound furan). For example, glucose's complete chemical name is glucopyranose, and the chemical name of fructose is fructofuranose. Sucrose's molecule consists of a glucose molecule with a six-member ring and a

fructose molecule with a five-member ring, so it is glucopyranosyl fructofuranose. (Note that in the case of a disaccharide's name, ***se*** in the first molecule's name changes to ***syl***).

- The **aldose** and **ketose** notations in the name of a sugar indicate its functional group. If the formula has an **aldehyde group** (HCO), the sugar is classified as an aldose (e.g., glucose and ribose). If it has a **ketone group** (CO), the sugar is classified as a ketose (e.g., fructose).
- The notations (+ or right hand) and (– or left hand) in the name of a sugar indicate the rotation of polarized light in polarimetry (see the Polarimeter subsection in Section 2 of Chapter 11). Right-hand sugars (e.g., sucrose, glucose, raffinose, dextran, and invert sugar) rotate polarized light to the right. Left-hand sugars (e.g., fructose) rotate polarized light to the left.
- The **alpha** (α) and **beta** (β) notations in the name of a sugar indicate the direction of the OH on the carbon atom that participates in glycosidic bonding. The glucose molecule is in α-form and fructose molecule is in β-form (see Figure 1.13), so the sucrose molecule is α-glucopyranosyl, β-fructofuranose.
- The notations D and L in the name of a sugar specify its type of isomer (two compounds with the same formula but different structures). D and L isomer in a sugar molecule is determined by the OH group attached to the farthest asymmetric (interior) carbon atom from the C–1 in its formula. When the OH is written to the right of this carbon, the D isomer is meant. When the OH is written to the left, the L isomer is represented. In glucose and fructose molecules, the farthest interior carbon atom from the C–1 is C–5. In both carbons, the OH groups in the straight method are written on the right, so both glucose and fructose are in D form (see Figure 1.13). (Note: Almost all carbohydrates are in D-family; when the notation is not given, the D-family is intended.)
- The notation of some numbers in front (or in the middle) of the name of a disaccharide, oligosaccharide or polysaccharide indicates the numbers of the carbon atoms that connect the rings through a **glycosidic** (oxygen) bond. In the sucrose molecule, the glycosidic bond connects C–1 (with aldehyde group) of the glucose ring to C–2 (with ketone group) of the fructose ring, creating a 1,2 glycosidic bond (see Figure 1.13). In raffinose, the glycosidic bond is 1,6-position (see Figure 1.20).

SUCROSE FORMATION

Sucrose is formed in many fruits (e.g., about 7% in bananas and 5% in oranges). Industrially, sugarbeet and sugarcane are the main sources of sucrose. Sucrose is formed by the **photosynthetic process** (see the Glossary). The primary product of photosynthesis is monosaccharides. Then, two monosaccharides combine with the help of an enzyme (named **sucrase**, not *sucrose*) to form sucrose (see Figure 1.14):

$$6CO_2 + 6H_2O + \text{Sunlight energy} \rightarrow C_6H_{12}O_6 + 6O_2$$

$$C_6H_{12}O_6 + C_6H_{12}O_6 + \text{Enzyme} \rightarrow C_{12}H_{22}O_{11} + H_2O$$

The second equation is called a **dehydration reaction** (condensation reaction) because one molecule of water is formed during reaction. The molecule of water formed during formation of sucrose occurs by the elimination of a molecule of water between the OH group on carbon 1 of the glucose unit and the OH group of carbon 2 on the fructose unit (see Figure 1.14). This is an 1:2 glycosidic bond (linkage). (Note: In the body, the dehydration reaction of sucrose

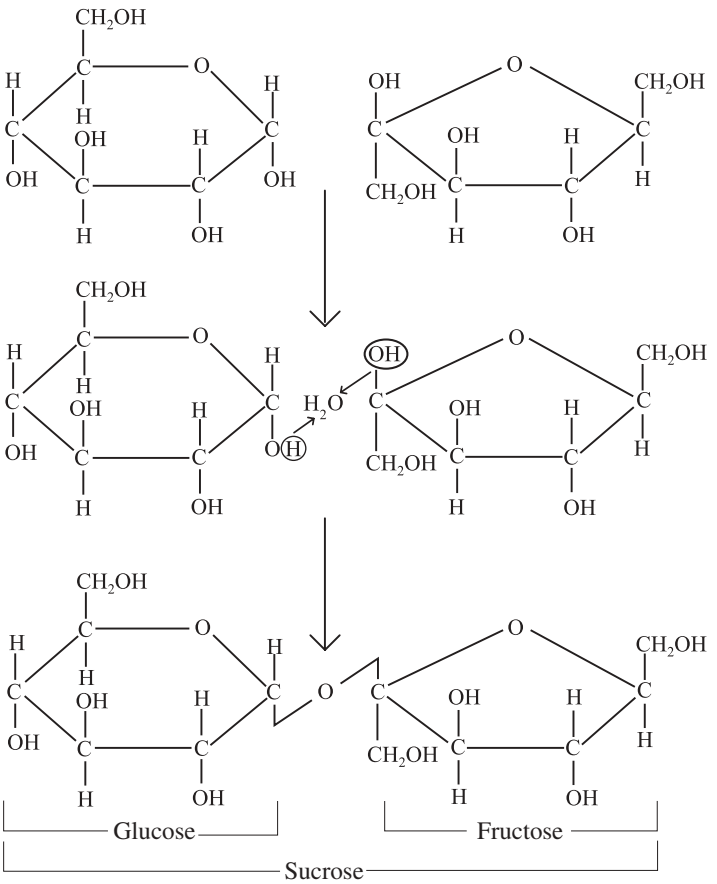


Figure 1.14 Sucrose formation

occurs by the **sucrase** enzyme. In the laboratory, it requires a dehydration agent such as sulfuric acid.)

Sucrose is found in many fruits (about 7% in bananas, 5% in oranges, and to a lesser extent in some vegetables). Sugarbeet and sugarcane are the main sources of sucrose. Sugarbeet contains 15 to 20% and sugarcane 10 to 17% sucrose. (Note: For more information on sugarbeet composition, see Chapter 3.)

SUCROSE DENSITY

Density (*d*) is the mass of a substance per its volume. The density of a sucrose crystal (refers to a single crystal, not in its bulk form) at 20°C is 1587.1 kg/m^3 (99.1 lb/ft^3).

Specific density is the ratio of the density of a substance to the density of water at the same temperature. Because specific density is a ratio of two numbers having identical units, it is **dimensionless** (without units). The density of water is 1 g/mL (at 4°C), so the specific density of a substance is numerically equal to its density.

Bulk density is the mass per volume of a *loose material*. Loose materials occupy more space than compacted forms because of empty space that exists between the particles. The

amount of extra space depends on the particle size (*the larger the sugar crystals, the larger is the empty space*).

The bulk density of sugar depends on its crystal size (*the finer the crystals, the larger is the bulk density of sugar*). The following table gives the approximate **bulk density** of fine, medium, and coarse sugars, cube sugar, and powdered sugar:

Fine sugar	880 kg/m^3 (55 lb/ft^3)
Medium sugar	860 kg/m^3 (54 lb/ft^3)
Coarse sugar	840 kg/m^3 (52 lb/ft^3)
Cube sugar	850 kg/m^3 (53 lb/ft^3)
Powdered sugar	650 kg/m^3 (41 lb/ft^3)

SUCROSE MELTING POINT

The **melting point** of a substance is the temperature at which it changes from a solid to a liquid. Sucrose is extremely stable in its crystalline (granulated) form at room temperature. Sucrose melts (turns into liquid) at higher temperatures of about 185°C, producing invert sugar and coloring substances.

As is seen in Figure 1.13, a sucrose molecule has eight **hydroxyl groups** (OH). In each group, the hydrogen atoms are slightly positive and the oxygen atoms are slightly negative. Therefore, sucrose molecule can form multiple **hydrogen bonds** (see Atom and Molecule subsection, in Section 1 of Chapter 12) with neighboring molecules. The hydrogen bonds are strong enough to make sucrose a solid at room temperature and give it a relatively high melting point. Sucrose molecules have a high melting point because of the high heat energy required to interrupt the hydrogen bonding and separate the molecules.

Caramelization is the process of decomposing sucrose to glucose, fructose, and finally to caramel (coloring substances) at temperatures close to its melting point (approximately 185°C). The product of the caramelization reaction is **caramel**, which has a brown color and a pleasant taste; caramel is used as a flavor enhancer in food products.

NOTE

Before leaving this topic, it may be helpful to clarify the difference between the dissolving process and the melting process with regard to *sucrose solutions*. Both turn sucrose crystals into a liquid, but each creates a different product. When sucrose crystals *dissolve* in water, a solution is produced in which the sucrose molecules and water molecules *remain intact*. In the solution, sucrose molecules move among the water molecules as well as among other sucrose molecules. If the water is allowed to evaporate from the solution, sucrose crystals form again. On the other hand, when sucrose crystals *melt*, their molecules decompose to glucose, fructose, and other substances. The sucrose molecules *do not remain intact* and will change to other substances.

SUCROSE SOLUTIONS' BOILING-POINT ELEVATION

The **boiling-point elevation** (*BPE*) of a solution is a solution's boiling point above that of pure water at the same pressure. The **boiling point** (*BP*) of a solution is the temperature at which the solution boils at atmospheric pressure (1 atm or 14.7 psi, the pressure at sea level). At atmospheric pressure (sea level), sucrose increases the boiling point of water to above 100°C. For example, a 50% sucrose solution boils at 101.8°C (*BPE* = 1.8°C).

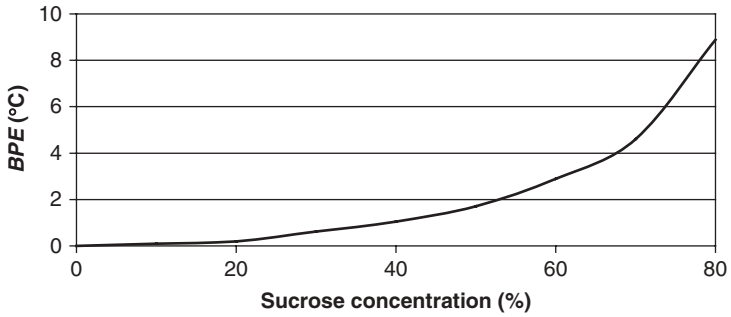


Figure 1.15 Effect of sucrose concentration on *BPE* at 90°C

The BPE of sucrose solutions increases as solution concentration increases (see Figure 1.15). During evaporation of thin juice, the *BPE* of the juice increases because water evaporates, resulting in an increase concentration of the solution (*as the dry substance of a solution increases, its BPE also increases.*)

As you will find in Chapter 12, **molality** (*m*, also known as **molal concentration**) is one of the ways of expressing the concentration of a solution. **Molality** (the number of moles of solute in 1 kg of solution) is useful for calculations of physical measurements such as *BPE*. A simple relationship exists between *BPE* (the amount of °C that the boiling point is raised) and *m* for *ideal solutions*:

$$BPE = K_{BP} \times m \tag{1.13}$$

K_{BP} Boiling-point constant of solvent (°C.kg/mole), for example, the K_{BP} for water is 0.51°C.kg/mole

m Molality (mole/kg)

$$BPE = °C \times \frac{kg}{mole} \times \frac{mole}{kg} = °C$$

NOTES

- **We cannot use the $K_{BP} = 0.51$ for high-concentration sucrose solutions and for impure sucrose solutions because these solutions deviate from *ideal solutions*. The K_{BP} for sucrose solutions depends on the purity of the solution and ranges from 1.9 (for a pure sucrose solution) to 2.5 for a 70% purity solution at 75°C. The followings are K_{BP} values for different juice purities (McGinnis 1982):**

Purity (%)	K_{BP} (°C.kg/mole)
100	1.9
90	2.0
80	2.2
70	2.5

- **The *BPE* of pure sucrose solutions is given in Table A.4 (in the Appendix).**

EXAMPLE 4

Given:

DS of a sucrose solution	15.0% (m/m)
Molecular mass (*MM*) of sucrose	342.3 (g/mole)

Calculate:
BPE of the solution

The 15.0% (m/m) sucrose solution contains 15.0 g of sugar in 100 g (0.1 kg) of solution. Eq. (12.11), given in Chapter 12, calculates the molality of the solution:

$$m = \frac{\text{Mass}_{\text{Solute}}}{MM_{\text{Solute}} \times \text{kg}_{\text{Solution}}} = \frac{15.0}{342.3 \times 0.1} = 0.44 \text{ mole/kg}$$

$$BPE = K_{BP} \times m = 0.51 \times 0.44 = 0.22°\text{C}$$

Because the boiling point of water at atmospheric pressure (1 atm = 1.013 bar = 101.3 kPa = 760 mm Hg = 760 torr = 14.7 psi) is 100°C, the boiling point of this solution is

$$100 + 0.22 = 100.22°\text{C}$$

SUCROSE SOLUTIONS' FREEZING-POINT DEPRESSION

The **freezing-point depression** (*FPD*) of a solution is the solution's freezing point below that of pure water. For example, a 50% sucrose (sugar) solution freezes at –7.6°C. Knowing that sugar lowers the freezing point of food products helps you understand why ice cream without sugar would freeze in the freezer so rock hard that it would be difficult to eat. In a similar manner, roads are salted during cold weather to decrease the *FPD*, thus preventing ice formation. In general, *the FPD decreases as the solution's concentration increases*.

The *FPD* of a solution is determined by an equation similar to the one for *BPE*:

$$FPD = K_{FP} \times m \tag{1.14}$$

K_{FP}	Freezing-point constant of solvent (°C.kg/mole), the K_{FP} for water is –1.86°C.kg/mole
m	Molality (mole/kg)

EXAMPLE 5

How much sugar must be added to 1.0 kg of water to lower the freezing point of the water from 0 to –2.0°C?

The **molality** (*m*) of a solution with *FPD* of –2.0°C can be found from Eq. (1.14):

$$m = \frac{FPD}{K_{FP}} = \frac{-2.0}{-1.86} = 1.08 \text{ mole/kg}$$

The mass of solute (sugar) can be determined by using Eq. (12.11) given in Chapter 12:

$$m = \frac{\text{Mass}_{\text{Solute}}}{MM_{\text{Solute}} \times \text{kg}_{\text{Solution}}}$$

$$\text{Mass}_{\text{Sugar}} = m \times MM_{\text{Sugar}} \times \text{kg}_{\text{Solution}} = 1.08 \frac{\text{mole}}{\text{kg}} \times 342.3 \frac{\text{g}}{\text{mole}} \times 1.0\,\text{kg} = 369.7\,\text{g}$$

SUCROSE SOLUBILITY

Sucrose is highly soluble in water. The reason for its high solubility is that sucrose contains eight free hydroxyl groups (OH) of which at least five can attach to other molecules depending on the molecule reactivity. For example, when sucrose is dissolved in water, each sucrose molecule can form at least five hydrogen bonds with water molecules. As the concentration of the solution increases, no more free water molecules are left in the solution for **hydrogen bonding** (see Section 2 in Chapter 12) and the solution becomes *saturated*. The **solubility** of a **solute**, such as sucrose, in a **solvent**, such as water, is the *maximum quantity of a solute that dissolves to produce a saturated solution at a certain temperature*. Solubility increases with temperature, as shown in Figure 1.16. For example, at 20°C, 2 parts of sucrose are dissolved in 1 part of water, but at 100°C (the boiling temperature of water), 4.76 parts of sucrose can be dissolved in 1 part of water to make 82.6% sucrose solution. (Table A.2 in the Appendix gives the sucrose solubility.)

The **polarity** (the property of molecules being **electrolyte** or **nonelectrolyte**) of molecules of the solute and the solvent determine whether the solute is soluble in the solvent (*solutes and solvents with similar polarity are soluble*). Based on this principle (known as the **like-dissolves-like** rule), a **polar** (electrolyte) solute is soluble in a polar solvent, but it is not soluble in a nonpolar solvent. A **nonpolar** (nonelectrolyte) solute is soluble in a nonpolar solvent, but it is not soluble in a polar solvent. With some exceptions, most solvents containing oxygen (O) are polar (see the Notes).

Sucrose (a nonpolar substance) is soluble in polar solvents, such as water, acetone (C_3H_6O), acetic acid (HCH_3COO), and formic acid (HCOOH). But it is not soluble in nonpolar solvents, such as chloroform ($CHCl_3$), carbon tetrachloride (CCl_4), hexane (C_6H_{14}), heptane (C_7H_{16}), toluene (C_7H_8), oil, and in a few other organic solvents. Sucrose is not soluble in isopropyl alcohol ($CH_3CHOHCH_3$) although it contains oxygen.

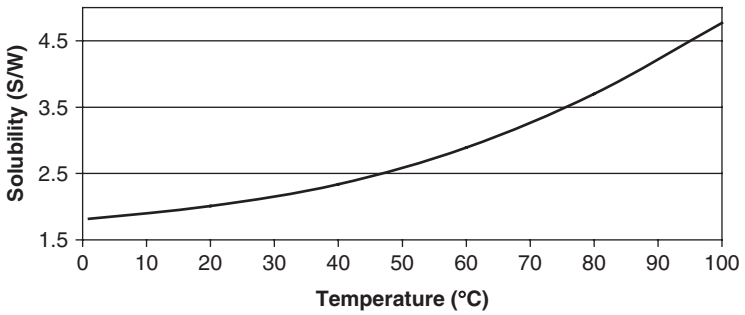


Figure 1.16 Sucrose solubility at different temperatures

SUCROSE IS NONREDUCING

Sucrose is a **nonreducing sugar** (unlike most other sugars) because its molecule does not have a free functional group in either of its two ring. In other words, the aldehyde group (HCO) in the glucose ring and ketone group (CO) in the fructose ring are blocked, giving the sucrose molecule the characteristic of a nonreducing sugar.

Nonreducing sugars cannot reduce the alkaline (basic) solutions of **oxidizing agents**, such as Fehling's, Benedict's, or Muller's reagent (alkaline-copper reagents) because neither of sucrose's rings can open for the reaction. On the other hand, **reducing sugars** (e.g., all monosaccharides, invert sugar, and most disaccharides and oligosaccharides) have a free functional group and therefore can reduce cupric ions (Cu^{2+}) of, for example, Fehling agent, to cuprous ions (Cu^{+}).

$$Cu^{2+} + 2e^{-} \rightarrow Cu^{+}$$

Sugar laboratories use the reducing property of **invert sugar** (the 50 : 50 solution of glucose and fructose produced from sucrose inversion) to determine its content in in-process products and molasses (see Section 7 in Chapter 11 for analytical procedure). In this test, invert sugar reacts with cupric ions (Cu^{2+}) of the oxidizing agent to precipitate cuprous oxide (Cu_2O) in the basic (alkaline) medium. Here, the aldehyde group on the reducing sugar is oxidized, and the Cu^{2+} ions of the oxidizing agent are reduced to Cu^{+} ions. The product is a brick-red precipitate of copper oxide (Cu_2O).

$$2C_6H_{12}O_6 + 4Cu(OH)_2 \rightarrow 2CH_2OH(CHOH)_4COOH + 2Cu_2O\downarrow + 4H_2O$$

(The $\downarrow$ sign represents precipitation.) With controlled conditions such as time, temperature, and reagents concentration, the amount of copper reduced is proportional to the amount of invert sugar in the sample.

SUCROSE IS NONIONIC

Nonionic compounds (also called nonpolar, molecular, or nonelectrolyte compounds) do not decompose (dissociate) to positive and negative ions in aqueous solutions. Therefore, they cannot conduct electric current. In the sucrose molecule, neither ring can open (decompose) in water. Sucrose and water are nonionic compounds; therefore, dissolving sugar in water does not improve its ability to conduct **electric current**.

As explained earlier, the sucrose molecule consists of a glucose and a fructose ring, connected by an oxygen (glycosidic) bond (see Figure 1.13). Because of the strength of **hydrogen bond** (see Section 1 in Chapter 12) between the sucrose rings, neither ring can open in water, so the sucrose molecule does not decompose. This property puts sucrose in the nonionic group.

Because nonionic compounds do not decompose when dissolved in water, their molecules stay intact in the solution. When sucrose dissolves in water, the crystals begin to dissolve but its molecules stay intact. This is because water molecules attract the sucrose molecules (because of high polarity of H_2O), forming **hydrogen bonds** between the molecules. As a result, the water becomes a **hydrated-sucrose solution** ($C_{12}H_{22}O_{11}.H_2O$) in which several water molecules surround each sucrose molecule. A molecule held in a group of water molecules are said to be in a **solvent cage** (see Figure 1.17). (Note: The number of water molecules in the solvent cage varies, depending on the concentration of the solution.)

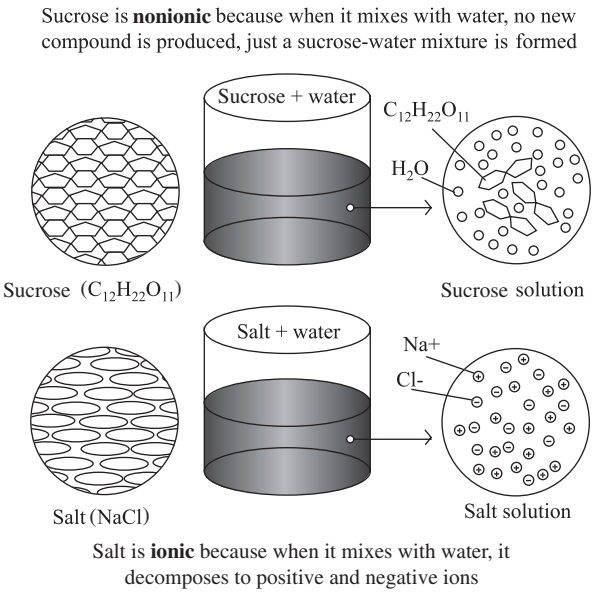


Figure 1.17 Sugar is nonionic and salt is ionic

Because the nonionic compounds keep their molecules intact in a solution, the change from solid form to liquid form is considered as a **physical change**. (The sucrose molecules in a solution are similar to molecules in a teaspoon of sucrose crystals.)

Ionic compounds (electrolyte compounds) decompose to positive and negative ions in an aqueous solution; therefore, they can conduct electric current. **Salt** (sodium chloride, NaCl) is an ionic compound because it decomposes to positive **sodium ions** (Na^+) and negative **chloride ions** (Cl^-) when dissolved in water. Here, none of ions has properties of the original substance. In other words, ionic compounds are different from the elements of which they are made (NaCl is different from sodium and chlorine). Therefore, the change is a **chemical change**.

NOTES

- **The properties of ionic and nonionic compounds are used in the molasses desugaring by chromatographic (MDC) process, an important process in the sugar industry. In this process, the *nonionic sucrose* in molasses is separated from *ionic nonsucrose substances* by using ion-exchange resins. Full information on molasses desugaring can be found in Chapter 8.**
- **The properties of ionic and nonionic substances are used in the conductometric determination of ash (refers to inorganic cations) content in sugar solutions. Ash in the sample decomposes to ions, causing the conductance of electric current, while sucrose does not carry electricity. (Refer to the Conductometer subsection in Section 2 of Chapter 11 for more information on analytical procedure for ash determination.)**

SUCROSE ACIDIC REACTIONS

Sucrose is instable in acidic solutions, decomposing to two simpler sugars: glucose and fructose. During this reaction, the acid (e.g., HCl) is a **catalyst** (a substance that speeds up a reaction

without being consumed). Actually, the reaction of sucrose in acid is the reaction of sucrose with water:

$$\text{Sucrose} + H_2O \rightarrow \text{Glucose} + \text{Fructose}$$

This type of reaction is called a **hydrolysis reaction** because one molecule of water is needed to break a reactant (here, sucrose) into two products.

Sucrose inversion is the reaction of sucrose with water in an acid solution or in the presence of an enzyme catalyst. The **inversion rate** (the measure of the change in concentration per unit time) depends on the following:

- Concentration
- Temperature
- pH

The amount of invert sugar formed (I) is proportional to the initial sucrose concentration (C_0) and time (t) during which the inversion taking place.

$$I = KC_0t \tag{1.15}$$

where K is an inversion constant that shows the quantity of invert sugar formed per unit of time ($t = 1$) from an initial sucrose concentration of $C_0 = 1$. K depends on the pH:

$$K = K_0(H^+) \tag{1.16}$$

K_0 is the constant, independent of the pH but dependent on the temperature. The following table gives the values of K_0 for different temperatures (Silin 1958):

Temperature (°C)	K_0
50	0.12
60	0.38
70	1.18
89	3.30
90	8.92
100	26.80

EXAMPLE 6

Calculate the amount of invert sugar produced from 100 parts of a sugar solution with pH = 5, heated for 45 minutes at 90°C.

$$\text{pH} = 5(H^+) = 10^{-5}$$

At 90°C, the K_0 is 8.92 and $K = K_0\,(H^+) = 8.92 \times 10^{-5}$

Initial concentration (K_0) is equal to 100 because the granulated-refined sugar is 100% sucrose.

$$I = KC_0t = 8.92 \times 10^{-5} \times 100 \times 45 = 0.4 \quad \text{parts or 40\% of total sugar}$$

Note that at pH = 4 (stronger acid solution than pH = 5), the invert quantity would be 10 times larger. As Figure 1.18 indicates, the same amount of inversion (I) is achieved when pH is decreased from 5 to 4 and the temperature from 90 to 70°C.

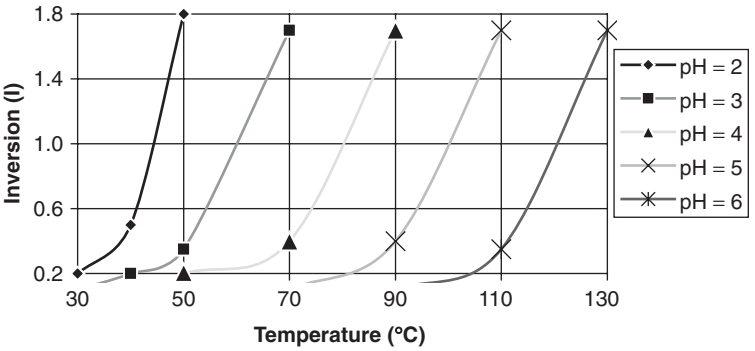


Figure 1.18 Sucrose inversion rate at different pH and temperature values

NOTES

- **In sugar terms, sucrose inversion during beet storage and beet processing is considered sucrose lost. Sugarbeet loses a considerable amount of its sugar during storage due to respiration (breathing) and microorganisms. This is not a welcome action in the sugar factory because it creates not only the sugar loss but also difficulties during the process, particularly in the crystallization station. In addition, the invert sugar, produced from sucrose inversion, decomposes to some acids and coloring substances that increase nonsucrose content. Therefore, sugar technologists try to reduce the rate of inversion in in-process products by keeping the pH of the solutions above 7. The least inversion occurs when the pH is between 7.5 and 8.5 (a weak alkaline media).**
- **The term sucrose degradation is also used by sugar technologists to denote sucrose inversion. This is because chemical degradation is defined as the gradual decomposition of a substance to another substances with a *lower degree of value*. Only the terms sucrose inversion and sucrose decomposition are used in this book.**

SUCROSE ALKALINE REACTIONS

Sucrose does not decompose in almost any alkaline solution. **Alkalis**, also known as **bases** (substances that produce hydroxide ions in solution) in alkaline solutions combine with sucrose (under certain conditions) to produce **saccharates** (see the Note below). During the alkaline combination, sucrose acts as a weak acid by releasing a hydrogen ion (H^+) that reacts with the hydroxide ion (OH^-) from the base to produce water and saccharate (see the following Note).

Sucrose reacts with lime [$Ca(OH)_2$] to form monosaccharate calcium ($C_{12}H_{22}O_{11}.CaO$), disaccharate calcium ($C_{12}H_{22}O_{11}.2CaO$), or trisaccharate calcium ($C_{12}H_{22}O_{11}.3CaO$), depending on the concentration and temperature of the solution. Monosaccharate and disaccharate calcium are soluble in water, but trisaccharate calcium is not.

NOTE

- **Saccharates (the salts composed of oxides with sucrose, e.g., calcium saccharate) and saccharides (the scientific name for sugars) are not the same.**

SUCROSE HALF-LIFE

The **half-life** ($t_{1/2}$) of a reaction is the time required for the concentration of a **reactant** (a starting substance in a chemical reaction) to decrease to half its initial value. Half-life is important in chemistry because it is constant in a first-order reaction such as sucrose inversion in acid solutions. Therefore, the half-life can determine the speed of a reaction. The half-life of a reaction ($t_{1/2}$) is determined by using the following equation:

$$t_{1/2} = \frac{\ln 2}{k} = \frac{0.693}{k} \tag{1.17}$$

k Rate constant (it depends on temperature; k for *sucrose* at 25°C is equal to 0.208 per hour)
ln Natural log (log to the base of e = 2.71828)

The inversion of sucrose to invert sugar is an **oxidation-reduction reaction**. Oxidation and reduction reactions go hand-in-hand (one cannot occur without the other). In other words, when one substance is **oxidized** (lose electron), another is **reduced** (gain electron). In an oxidation-reduction reaction, two reactions take place simultaneously, representing one process called a **half-life reaction**. So the half-life refers to the time that the concentration of a reaction is decreased to half its initial value.

EXAMPLE 7

What amount of time is required for sucrose to invert to a 50% invert sugar in an acidic solution at 25°C?
Because sucrose is 100% pure, its inversion to 50% corresponds to the definition of the half-life reaction, so Eq. (1.17) can be used.

$$C_{12}H_{22}O_{11} + H_2O \rightarrow C_6H_{12}O_6 + C_6H_{12}O_6$$

$$t_{1/2} = \frac{0.693}{k} = \frac{0.693}{0.208} = 3.3 \text{ h}$$

Note: The preceding calculations are based on approximate values and in reality the reaction time is different. Usually, the sugar industry produces 50% **invert liquid** at 70°C at pH of about 4.5, and the process takes about 30 minutes to complete.

SUCROSE SPECIFIC HEAT

Specific-heat-capacity (C_P, often called simply **specific heat**) is the amount of heat needed to raise the temperature of 1 kg of a substance by 1°C, *without a change in state*. The heat capacity is expressed in kJ/kg.°C (reads kilojoules per kilograms per degrees Celsius).

Not all substances have the same temperature increase when an equal amount of heat energy is added. In other words, different materials have different heat capacities. The specific-heat-capacity of water is 4.187 kJ/kg.K or 1 Btu/lb.°F (see the following Note). This means that for each degree Celsius that water is heated, a kilogram of water absorbs 4.187 kJ of thermal (heat) energy. Or, put another way, it takes 418.7 kJ energy to heat 1 kg of water from 0 to 100°C.

The specific heat of pure and impure sucrose solutions decreases when their concentration and temperature are increased. You can calculate the specific heat of pure and impure sucrose solutions by using the following equation (Bubnik et al. 1995):

$$C_P = 4.187 - DS \times (0.0297 - 4.6 \times 10^{-5} \times P) + 7.5 \times 10^{-5} \times DS \times T \qquad (1.18)$$

DS	Dry substance content (for pure sucrose solutions, $DS = S$)
T	Temperature (°C)
P	Purity (for pure solutions, $P = 100$)

EXAMPLE 8

Given:

Sugar content (S) of a pure sucrose solution	50.0%
Temperature (T)	60°C

Calculate:

Specific heat (C_P) of the solution

$$C_P = 4.187 - 50.0 \times (0.0297 - 4.6 \times 10^{-5} \times 100) + 7.5 \times 10^{-5} \times 50.0 \times 60) = 3.16$$

NOTES

- **The value of kJ/kg.°C is equal to kJ/kg.K, because a 1° change in temperature is the same on the Celsius and Kelvin scale.**
- **The specific heat of *impure sucrose solutions* is slightly lower than that of *pure sucrose solutions* (the specific heat of impure sucrose solutions decreases as purity decreases) at the same dry substance and temperature.**
- **The specific heat of *pure sucrose solutions* is given in Table A.6 and that of *impure sucrose solutions* is given in Table A.7 (in the Appendix).**

SUCROSE SPECIFIC ENTHALPY

The **specific enthalpy** (H, often shortened to **enthalpy**) is the amount of **heat content** of a solute (the dissolved substance) during a chemical reaction at constant pressure. The enthalpy change (ΔH) is the difference between the final and initial **enthalpy** (the heat change during a chemical reaction) contents. The amount of **heat** (heat energy) released or gained by a chemical reaction can be measured by the change in enthalpy. Because the reaction is carried out at constant pressure, the heat released or gained (Q) is the same as the enthalpy change (ΔH).

The quantity of heat transferred during a chemical reaction depends on the amount of **reactant** (the starting substance in a chemical reaction) used or **product** (the finishing substance in a chemical reaction) produced. The enthalpy is expressed in kilojoule (kJ) or kilocalorie (kcal) per mole or per kg of a reactant (or product). For example, one mole of sucrose (reactant) is oxidized in the human body to carbon dioxide and water (products) to release 5645 kJ of energy (H, heat content):

$$C_{12}H_{22}O_{11} + 12O_2 \rightarrow 12CO_2 + 11H_2O \qquad \Delta H = -5645\,\text{kJ}$$

This type of reaction is called an **exothermic reaction** because during the reaction, energy in the form of heat is produced. Here, each mole of sucrose (342.3 g) produces 1349.2 kcal (equal to 5645 kJ) of **heat** (thermal energy). In other words, the energy released (Q) by this reaction is equal to 5645 kJ/mole (1349.2 kcal/mole).

EXAMPLE 9

Given:

One serving size of sucrose (molecular mass, MM = 342.3 g 4 g

Calculate:

Energy released (H, heat content) by a serving size of sugar (sucrose)

Number of mole of sucrose is found from (Eq. 12.3) in Chapter 12,

$$\text{Mole} = \frac{\text{mass}}{MM} = \frac{4}{342.3} = 1.169 \times 10^{-2}$$

$$H = 1.169 \times 10^{-2} \text{ mole sucrose} \left(\frac{-1349.2 \text{ kcal}}{1 \text{ mole sucrose}} \right) = -15.8 \text{ kcal/4g } (-66.1 \text{ kJ/4g})$$

You can calculate the specific enthalpy (in kJ/kg) of pure and impure sucrose solutions using the following equation (Bubnik et al. 1995):

$$H = T \lfloor 4.187 - DS(0.0297 - 4.6 \times 10^{-5} \times P) + 3.75 \times 10^{-5} \times DS \times T \rfloor \qquad (1.19)$$

DS	Dry substance content (for pure sucrose solutions, $DS = S$)
T	Temperature (°C)
P	Purity (for pure solutions, $P = 100$)

EXAMPLE 10

Given:

DS of an impure sucrose solution	50.0%
P	90.0%
T	60°C

Calculate:

Specific enthalpy (H) of the solution (in kJ/kg)

$H = 60 \lfloor 4.187 - 50.0(0.0297 - 4.6 \times 10^{-5} \times 90.0) + 3.75 \times 10^{-5} \times 50.0 \times 60 \rfloor = 181.3$ kJ/kg

NOTES

- **The energy released in the preceding and similar reactions can be measured in a calorimeter. (A calorimeter can measure the heat released or absorbed in a reaction by using the temperature difference from oxidizing a known amount of reactant.) Using mass (m), specific heat capacity (C_p), and temperature change ($T_2 - T_1$) as variables, you can calculate the heat transfer (absorbed or released) by a reaction (Q) by the following equation:**

$$Q = C_P \times m(T_2 - T_1) = m \times H \quad (1.20)$$

- **The specific enthalpy of impure sucrose solutions is slightly lower than that of pure sucrose solutions at the same dry substance content and temperature.**
- **The specific enthalpy of pure sucrose solutions is given in Table A.8 and that of impure sucrose solutions is given in Table A.9 (in the Appendix).**

SUCROCHEMISTRY

Sucrochemistry is the study of using sucrose as a raw material for production of other products. Sucrose with its 8 free hydroxyl groups (OH) has the potential to react with various functional groups, so it reacts with other substances to produce other products. Such reactions include sucrose alkylation (substitution of alkyl group [C_nH_{2n+1}] with hydroxyl groups [OH] of sucrose), sucrose estrification (substitution of ester group [C_nH_{2n+1}] with hydroxyl groups of sucrose), and sucrose etherification (substitution of ether group [R–O–R] with hydroxyl groups of sucrose to produce ether derivates e.g., sucrose octa-methyl-ether, which is used in safety glasses). These are part of the programs to expand the use of sucrose as a biomass (raw material from plants and animals). Many sucrose-based products are available on the market, such as the following:

- Bioethanol
- Low-calorie fat
- Bulking ingredients
- Low-calorie sweeteners
- High-intensity sweeteners
- Surfactants (e.g., detergents)
- Emulsifiers (e.g., skin moisturizers)
- Carbohydrate-based packing material (called peanuts)

Bioethanol (ethanol produced from plant sources) produced from sucrose, molasses, or sucrose-containing raw materials (mainly sugarcane) is the most important nonfood product of sucrose. Ethanol-based fuel is considered as the gasoline of the future. Because of a recent steady increase of oil price, every country tries to reduce its dependence on oil-based fuel. In the United States, a program called 25/25 aims to get 25% of its energy from renewable resources (e.g., ethanol, solar, and wind) by the year 2025. Ethanol produced from sugarcane and corn plays an important role in this program.

Bioethanol is produced in countries such as Brazil, the United States (ethanol is produced from corn), India, Pakistan, and some European countries. It is mixed up to 85% with gasoline as a blend of fuel for most cars, or it is used without mixing in specially designed cars. Bioethanol consumption counts for 2.5% of the world gasoline use (Henrique et al. 2005).

In the world, Brazil leads sugar and ethanol production, both of which come from sugarcane. In Brazil, ethanol is produced from sugarcane juice, molasses, or mix of both (Henrique et al. 2005) with an expected production of about 17.5 million m^3 in 2005/2006 campaign. As a result, Brazil became an energy-independent nation and its sugar surplus was reduced. Ethanol produced in Brazil costs less than U.S. ethanol produced from corn.

European countries have become interested in ethanol production from sugarbeet. France produces 125000 m^3 ethanol ($d_{Ethanol}$ = 800 kg/m^3) a year from sugarbeet, wheat, and maize. Bioethanol production helps in the fuel balance worldwide. Unlike fossil fuel, bioethanol production is environmentally friendly. In some countries, the government provides tax exemptions for bioethanol producers; in Canada, for example, the exemption is 10 cents per liter (Zucker Industrie, February 2005).

Low-calorie fat (fat replacer) is a generic term for ingredients that replace fat. These food products have carbohydrates as more than half their ingredients. Olestra, a fat replacer developed by Proctor & Gamble, is synthesized from sucrose and vegetable oil. It has physical properties similar to conventional fats. Olestra passes through the body undigested because of its molecular complexity and therefore adds no fat or calories to food. In 1996, the FDA approved Olestra for use in salty snacks.

Carbohydrate-based packing materials (peanuts) were developed to be environmentally friendly. Although they have many of the properties of polymer, they dissolve in water, thus reducing the landfill burden. Research is also underway to develop polymers that degrade in sunlight.

Sucrose is also used for the production of products such as **acetic acid** (HCH_3COO), **dextran** (a polysaccharide used in pharmaceutical and photographic industry), and some other polysaccharides. Dextran is produced through a fermentation process.

NOTE

Glycobiology is a new branch of biology that studies the role of sucrose and other carbohydrates in the production of new products through biochemistry. This science is based on the vast possibilities in the combination of simple sugars. For example, researchers are paying attention to the cell-coating property of sugars when cells suffer an infection because sugars can make chemical bonds with protein molecules of the damaged cells to prevent them from spreading.

CHAPTER 1

SECTION 5

CARBOHYDRATES

SUBSECTIONS

- Monosaccharides
- Disaccharides
- Oligosaccharides
- Polysaccharides
- Food Pyramid

ABOUT THIS SECTION **This section** discusses carbohydrates, particularly one of the subgroups: saccharides (sugars). Sucrose (sugar) also belongs to this group, but because its properties were discussed in detail in the previous section, here the focus will be on the other types of saccharides.

It is easy to define carbohydrates chemically, but it is difficult to do so from the point of view of an ordinary person. This is because of the confusion surrounding the amount of carbohydrates we should eat with our daily food intake. All nutritionists believe that some carbohydrates must be included in our daily food, but they do not agree on the amount. This section does not have an answer to this dilemma. Instead, it focuses on the properties of carbohydrates from the sugar-technology perspective.

Carbohydrates exist in major food products such as bread, rice, pasta, sugars, potatoes, fruits, and vegetables (see Figure 1.19).

Carbohydrates (from *carbon hydrates*) are saccharides, starches, and dietary fibers. Carbohydrates are a large class of substances with similar structures. They consist of carbon (C), hydrogen (H), and oxygen (O). Most of the carbon atoms in carbohydrates molecules are bonded to one hydroxyl group (OH) and one hydrogen atom (H). These carbon atoms are called interior (asymmetric) carbons. Some carbon atoms are bonded to a functional group (e.g., aldehyde or ketone functional groups).

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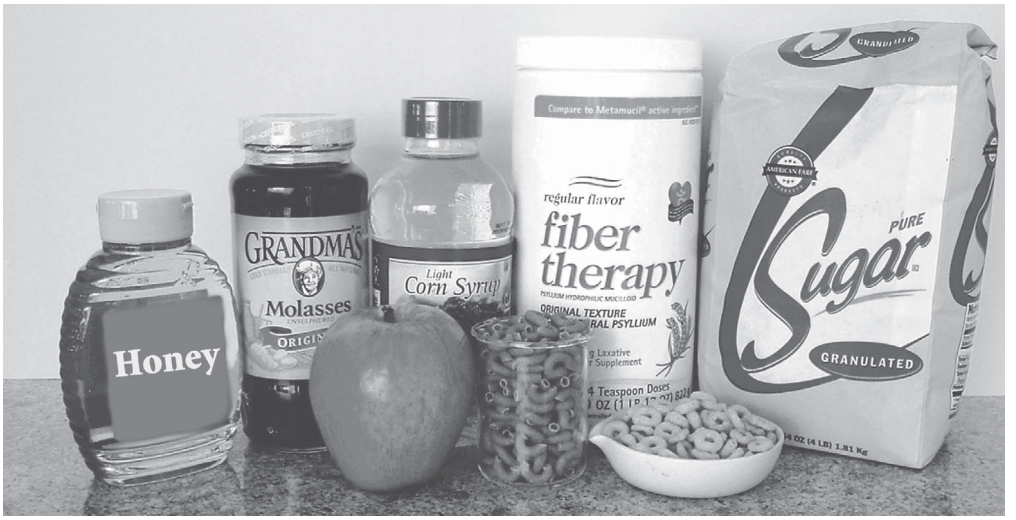


Figure 1.19 Some carbohydrates: Honey, molasses, apple, corn syrup, pasta, fiber, cereal, and sucrose

The quantity of hydroxyl groups (OH) in saccharides contributes to their sweetness. **Glucose** ($C_6H_{12}O_6$) with five hydroxyl groups and **sucrose** ($C_{12}H_{22}O_{11}$) with eight hydroxyl groups are sweet. In general, compounds with at least two hydroxyl groups on adjacent carbon atoms have sweetness. Glycerol (glycerin, $C_3H_8O_3$)with three OH groups, xylitol ($C_5H_{12}O_5$)with five, and glucitol (sorbitol, $C_6H_{14}O_6$) with six OH groups are less sweet than sucrose. Xylitol and glucitol are used in sugarless gum because bacteria in the mouth do not break them down into the **carboxylic acids** (acids containing COOH groups) that cause tooth decay. However, both are metabolized by the body in the same way as sugars (they break down to glucose).

In medical science, sugars are rated according to how quickly they increase blood glucose level relative to glucose. In this rating, called the **glycemic index** (GI), glucose has a standard value of 100, whereas the GI of sucrose is 64, which is relatively low. Sucrose has a low GI because it contains a molecule of fructose, which is very low on the glycemic index (Hannah 1998).

MONOSACCHARIDES

Monosaccharides are the simplest carbohydrates consisting of one unit, so they cannot be further decomposed to a simpler sugar. They have three to seven carbon atoms, and each carbon has one aldehyde (HCO) or ketone (CO) group. Those with aldehyde group (e.g., glucose) are known as **aldoses** and those with ketone group (e.g., fructose) are **ketoses**.

Glucose (dextrose) and **fructose** (levulose) are the common monosaccharides, with formulas $C_6H_{12}O_6$. They are six-carbon sugars and have the same formula but different structures, so they are **isomers**. Glucose is an aldohexoses, and fructose is a ketohexoses (see Figure 1.12 and 1.14). Because glucose and fructose promote the growth of microorganisms in the fermentation process, they are called **fermentable carbohydrates**.

Glucose (blood sugar) is one of the most abundant organic compounds on earth. Glucose exists in some fruits such as grapes, figs, and dates. It is the building block for molecules of two important polysaccharides: starch and cellulose.

Fructose (fruit sugar) is found in honey and many fruits such as apple and grapes. Industrially, it can be produced from sucrose or inulin (a polysaccharide). In **beet juice**, glucose and fructose are the main monosaccharides. Beet juice always contains more glucose than fructose.

Invert sugar is mixture of equal parts (50 : 50) of glucose and fructose. Invert sugar has greater sweetening power than an equivalent amount of sucrose because fructose is much sweeter than sucrose and glucose is slightly less sweet than sucrose. During the crystallization process of beet juice, invert sugar has less tendency to crystallize than sucrose.

The aldehyde (HCO) or ketone (CO) functional group of monosaccharides can be oxidized (by mild oxidizing agents, such as Fehling agent) or reduced (by reducing agents e.g., H_2). The reactions occur at C–1 (carbon 1). The products of the oxidation process are **carboxylic acids**, so monosaccharides are **reducing sugars**. The carboxylic acids formed are named by changing the *–ose* ending to *–onic acid*. For example, glucose produces gluconic acid.

In reduction process, the carbonyl group (aldehyde or ketone) reduces to hydroxyl (OH) to produce **sugar alcohols** (polyols). For example, xylose yields xylitol. Some sugar alcohols (e.g., xylitol and sorbitol) are used in food products to provide low sweetness. Because of hydroscopic property, some sugar alcohols are used in cosmetic products as **moisturizing agents**.

DISACCHARIDES

Disaccharides are carbohydrates composed of two monosaccharides, so they can be hydrolyzed to two monosaccharides. For example, sucrase (an enzyme) hydrolyses the glycosidic (oxygen) bond in the sucrose molecule to produce two monosaccharides (glucose and fructose). **Sucrose** (sugar), **lactose** (milk sugar), and **maltose** (malt sugar) are the common disaccharides, with formulas $C_{12}H_{22}O_{11}$. Unlike sucrose, lactose and maltose are **reducing sugars**.

OLIGOSACCHARIDES

Oligosaccharides (from the Latin *oligo* meaning **a few**) consist of three to six monosaccharides. Therefore, trisaccharides up to hexasaccharides are in the oligosaccharide group.

Raffinose ($C_{18}H_{32}O_{16}$) is a trisaccharide consisting of one molecule of galactose joined to a molecule of sucrose by an alpha 1 : 6 glycosidic (oxygen) bond (see Figure 1.20).

In sugarbeet, the raffinose content can vary largely depending on the location. In healthy beets, the raffinose content ranges from 0.2 to 0.5%. Raffinose is also found in beans, onions,

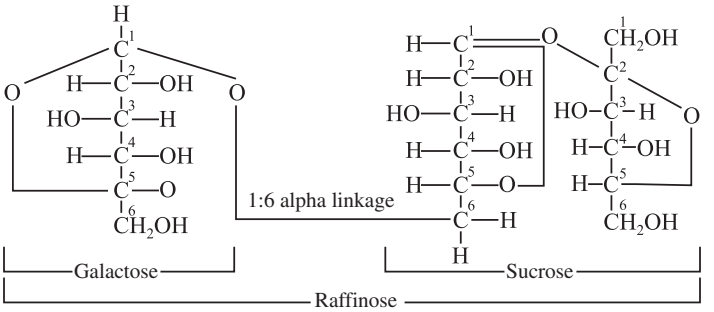


Figure 1.20 Raffinose configuration

and some other plants. Like sucrose, raffinose is a nonreducing sugar. Unlike sucrose, raffinose has very little sweet taste. Industrially, raffinose can be produced in a special method of the molasses-desugaring process by chromatographic (MDC) process (the special method for Raffinose is known as the KAAK process) as a separate fraction (van der Poel et al. 1998).

Stachyose is tetrasaccharide and verbascose is a pentasaccharide, both found in beet juice in trace amounts.

POLYSACCHARIDES

Polysaccharides (complex carbohydrates) are long molecules that consist of many repeating monosaccharides (hundreds to thousands) connected by alpha 1 : 2 glycosidic bonds. They are produced by consecutive **dehydration reactions** (losing-water reactions) of monosaccharides. Eventually, a polysaccharide of several thousands of saccharides can be obtained. Note that under suitable conditions, the dehydration reaction can continue indefinitely because the terminal units always have one or more unreacted OH groups. Polysaccharides are **nonreducing** sugars.

Starch is the best-known polysaccharide, consisting of 200 to 6000 glucose units. Unlike many other saccharides (sugars), starch itself is not sweet. If we hold a piece of bread in our mouth for a few minutes, it begins to taste sweet, indicating that digestion has begun and glucose is formed. In the body, starch is broken down into glucose and enters the bloodstream to become available to generate energy. Major sources of starch are potatoes, wheat, rice, and beans.

Starch is a glucose polymer, joined by alpha 1 : 4 glycosidic bonds (see Figure 1.21). Plants produce two forms of starch: amylopectin and amylose. Amylopectin accounts for about 80% of starch, has larger molecules (up to 100000 units) than amylose, and is not water-soluble. Amylose accounts for about 20% and is soluble in hot water.

In the **iodine test**, an iodine reagent (potassium iodide solution) is used to detect traces of starch. In this test, a drop of reagent added to a sample containing starch creates a purple color.

Pectin and dietary cellulose present in fruits are fibrous polysaccharides. These are used in our foods as dietary fibers, which can lower the cholesterol level in the blood because of their interaction with **bile salts** (cholesterol-derived substance produced in the liver).

Pectin is a fibrous polysaccharide that is insoluble in cold water but becomes gradually soluble in boiled water. It is made up of many galactose units. Sugarbeet contains 2 to 3% of pectin in its pulp (marc). Pectin exists in some vegetables and fruits. Pectin can be extracted from fruits and used in jam and jelly because it acts as a **gelling** (thickening) **agent** (becomes a gel when dissolved in a small amount of water). Sugarbeet pectin has lower gelling power

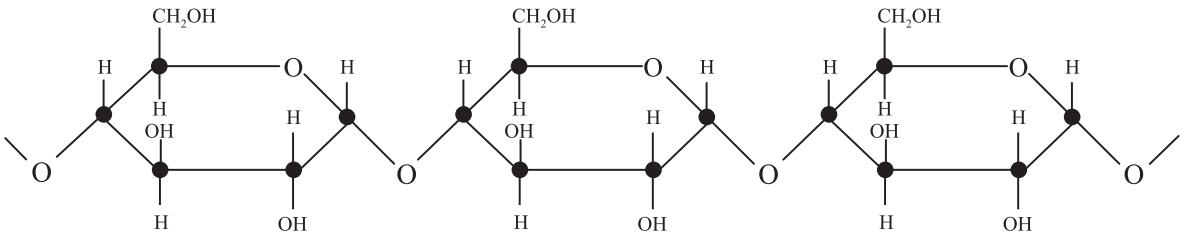


Figure 1.21 Starch configuration

than apple or citrus pectin, because the molecular mass of sugarbeet pectin (15000 to 50000) is smaller than apple or citrus pectin (70000 to 90000).

Cellulose is an insoluble fibrous polysaccharide that makes the structure in plants (cellulose forms about 50% of the woody parts of plants). It is a straight-chain polymer, consisting of 5000 to 10000 molecules of glucose. The glucose molecules are joined by beta 1 : 4 glycosidic (oxygen) bond. Cellulose is not water-soluble. We cannot digest cellulose because we do not have an enzyme that can hydrolyze the beta-glycosidic chain in cellulose. Animals, however, can digest cellulose because of having special enzymes (e.g., arabinase and pectinase) in their digestive system.

Dextran is a long-chain gelatinous water-soluble polysaccharide made up of glucose units. Dextran belongs to the colloid family, so it has damaging effects on sugarbeet processing (see Section 7 of Chapter 3). Dextran is used in pharmaceutical and photographic industry. Industrially, dextran is produced from sucrose by a fermentation process.

FOOD PYRAMID

The **Food Guide Pyramid** in Figure 1.22 is an effort from the U.S. Department of Agriculture (USDA) to show people how to eat healthily. No single food has all the nutrients that our body needs. The food pyramid can help people choose balanced foods (foods that include most of the nutrients that our body needs). In other words, the food pyramid instructs us to eat less of some foods and more of others.

The FDA recommends that packaged food products carry standardized **nutritional facts** on labels. The recommended nutritional facts are 300 g of carbohydrates and 25 g of dietary fiber per day. These quantities provide 1200 Calories (or kcal with a small **c**) a day, which accounts for 60% of the total daily recommended diet of 2000 Calories.

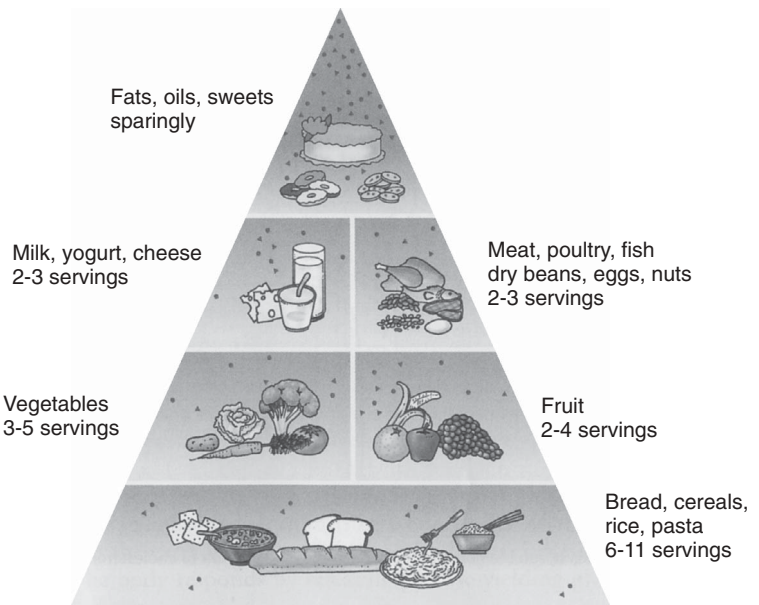


Figure 1.22 Food pyramid

END-OF-THE-CHAPTER NOTES

- In **Chapter 1**, readers unfamiliar with the sugar industry learned general information about sugar, the sugar factory, sugar terminology, and other related subjects. Readers familiar with the sugar industry have refreshed their memory and could have learned a few things they had not known before. With this introduction, you are in a good position to understand better the rest of the book. You could use this chapter as a reference while studying the rest of the book.
- In **Chapter 2**, we shift our attention to the topic of sugarbeet production in the farm.

CHAPTER 2

SUGARBEET FARMING

SECTIONS

- Soil Preparation
- Sugarbeet Planting
- Sugarbeet Fertilizing
- Sugarbeet Growth and Quality
- Sugarbeet Harvesting
- Sugarbeet Yield
- Sugarbeet Seed Production
- Sugarbeet Diseases and Insects
- Sugarbeet and Genetic Engineering
- Organic Sugarbeet
- Sugarbeet and Natural Environment
- Beet Laboratory and Growers' Payment
- Sugarbeet Farming Problems

ABOUT THE CHAPTER **Chapter 2** discusses how quality sugarbeet is grown in the field. Toward this goal, the focus will be on farming issues and problems that sugarbeet growers face in their routine work.

"*Sugar is made in the field, not in the factory.*" This statement expresses the goal of the beet growers who try to cooperate with the processor to produce sugarbeet with the highest possible quality. Here, high quality refers to sugarbeet with high-**sugar** (sucrose) and low-**nonsugar** (nonsucrose) content with the lowest possible **tare** (impurities). Cooperation between beet growers and processors is beneficial to both parties because the growers are entitled to a sufficient profit, and the processors have to be secure in the maket. In **cooperatives** (see Section 1 in Chapter 1), growers are in charge of both elements: beet growing and its processing.

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Figure 2.1 Sugarbeet farm



Figure 2.2 Sugarbeets ready for processing

Sugarbeet (see Note on page 2 of the book) belongs to the spinach family. In ancient times, sugarbeet was grown in Greece for its edible leaves. Around the tenth century, some varieties of sugarbeet were brought to Russia from Greece. This is the reason for the similarity in the word for sugarbeet in both countries: *svekla* in Russian and *svekle* in Greek (Silin 1958).

Sugarbeet (simply beet) grows in moderately cold climates but can adapt itself to very cold and warmer climates as well. In Europe, it grows almost everywhere, from the southern temperate country of Turkey to the northern cold countries of Sweden, Denmark, and Finland. The tropical sugarbeet varieties developed in Italy and Spain grow well in saline and alkaline soil under hot conditions (see the following Notes). Unlike sugarbeet, sugarcane grows in tropical areas. Only a few countries in the world (United States, Spain, Iran, Egypt, Morocco, and Pakistan) have the right conditions for growing both crops.

There are about 50 beet-growing countries in the world. Sugarbeet grows in most European countries, including the 25 countries in the European Union. In the United States, sugarbeet is grown in 12 states (California, Oregon, Idaho, Utah, Montana, Wyoming, Colorado, North Dakota, South Dakota, Nebraska, Minnesota, and Michigan). Sugarbeet-growing regions in the United States are divided into three geographic areas. The eastern regions (Michigan and the Red River Valley of Minnesota and North Dakota) practice **nonirrigated farming** (because they have enough rain), the western regions use **irrigated farming**, and the mid-regions use both systems depending on the amount of rain. The Red River Valley of Minnesota and North Dakota rank as the most advanced in sugarbeet cultivation. In Asia, sugarbeet is grown in Iran, Israel, Lebanon, China, Korea, Japan, the northern part of Pakistan, and a few other countries. In Africa, the northern part of Morocco and Egypt grow sugarbeet.

Sugarbeet is a **biennial plant** (a plant requiring two years for a full life cycle). In the first year of growth, the root (the part containing sucrose) and leaves grow. In the second year, the sugarbeet flowers and produces seed. (Note: Only the sugarbeet roots grown in the first year are used for sugar production.) Therefore, to produce sugar, the sugarbeet is treated as an annual. Seed is planted in the spring, and sugarbeet is harvested in the late fall or early winter. Typically, sugarbeet needs between six to eight months to grow and become ready for processing in the factory. In hot climate areas, sugarbeet is planted in the fall and harvested the next summer because of the mild winters.

Sugarbeet is a root crop and it stores sucrose in its root. When fully grown, it weighs 0.5 to 2 kg (about 1 to 4 lb). Typically, sugarbeets produce sugar (granulated-refined sugar) equal to one-seventh of their weight after sugarbeets are processed in the factory. The typical sucrose content for mature sugarbeets is 17%, but the value depends on the variety, year-to-year variation, and growing conditions. The sugar yield per hectare (ha) planted (**extractable sugar per acre**) from beets is about 7 tons per ha (3 short tons per acre). This value for sugarcane (usually contains about 12 to 17% sucrose) is 10 t/ha (4.4 short tons per acre). Note that the higher value for sugarcane is because of the much higher yield of sugarcane (about 100 t/ha), compared with about 40 tons of sugarbeet per hectare (17.5 short tons per acre). In the United States, nonirrigated areas can yield from 17 to 20 short tons sugarbeet per acre (39 to 45 metric tons per hectare), and the irrigated areas can yield from 30 to 45 short tons per acre (68 to 102 t/ha). In advanced sugarbeet-growing countries in Europe, the typical average **sugarbeet yield** is 50 to 60 t/ha (22 to 26 t/acre).

NOTES

- **Tropical sugarbeet (winter sugarbeet) developed in hot climate countries (e.g., countries in the south of Europe, north of Africa, and some parts of Asia) grow well in hot conditions and relatively do not need more water than beets grown in moderate climate countries.**
- **In the southern part of Iran, sugarbeet and sugarcane are grown side by side in irrigated farms under the same hot conditions. The tropical beet varieties are tolerant to the root and leaf diseases and**

are salt tolerant. Besides breeding, the most important factor for successful tropical beet growing is a locally adapted technique. The tropical sugarbeet takes between five to eight months to grow under a sufficient fertilization and irrigation schedule. A beet factory in the Sudan or in Pakistan can be in operation up to 300 days a year. In these areas, beet delivery to the factory is based on a strict schedule, arranged by the farmers and the factory (Nilsson 2005).

In the United States, a sugar factory with an average capacity of 10000 t beet-processing per day has about 250 growers, producing about 1.5 Mt beets per season in about 75000 acres (30000 ha) of land. This equates to a beet yield of about 20 t/acre (45 t/ha). (Note: The beet yield per acre in irrigated areas is higher.) The U.S. beet production is about 30 million t/year.

SOIL PREPARATION

Sugarbeet grows mainly in sandy loam, clay loam, and peaty loam soil. These types of soil (particularly with a mild slope) hold water better. Soil is a mixture of sand, silt, and clay. Sand contains the largest particles and clay, the smallest. In nature, soil usually contains three layers:

- **Topsoil:** It lies on the surface, and its depth is from a few centimeters to a couple of meters below the surface. Topsoil usually consists of equal amounts of sand, silt, and clay. Plant roots absorb most of their nutrients from topsoil.
- **Subsoil:** It lies under the topsoil; its height might be up to 1 m (3 ft) thick, and it often consists of clay. The roots of some plants reach the subsoil.

Soil preparation, such as tillage, seed spacing, fertilizing, and herbicide application are extremely important to obtain maximum production.

Tillage is the process of improving soil properties by **plowing** (soil turn-over). It is often performed in two stages. Secondary tillage is usually performed at shallower depths than primary tillage. Tilling helps agriculture in the following ways:

- Increases the respiration rate
- Increases the penetration of water
- Aerates the soil by loosening the soil
- Kills weeds by putting them under the soil
- Increases soil nutrients from decomposing plant material

The **percolation rate** (the flow of water through soil) depends on the soil compactness and its particle size. (*The less compact the soil, the greater is the percolation rate.*) Sugarbeet yield is sensitive to compaction. If the soil is compact, the percolation rate is low; the topsoil becomes **waterlogged**, preventing the plant from getting oxygen. If the soil is too loose, the percolation rate is excessively high; the soil becomes **leached**, causing water to flow quickly and removing many water-soluble nutrients, which are important to plant growth. The amount of water that a plant can access is important to the plant's growth. Sugarbeets grown in irrigated areas and in nonirrigated with enough rain have good-size roots and better sugar content. In some areas, such as the southern European countries, western United States, and some Asian areas, irrigation is necessary to grow sugarbeet.

Soil preparation for a sugarbeet crop usually begins in the fall by tilling. Primary tillage is deep and is commonly followed by secondary tillage, which is shallow.

Plowing of 20 to 30 cm (8 to 12 in.) deep usually results in the best sugarbeet crop. Moldboard plowing buries weed seeds and diseases. There is less disease when the previous crop and weed residue is buried. Shallow tillage with a chisel plow does not bury all residue but is better environmentally because the residue left on the surface after chisel plowing helps prevent wind and water erosion. Spring tillage with a cultivator is done just before planting to a depth of 8 to 10 cm (3.2 to 4 in.). (The shallower the spring tillage, the less drying of soil occurs.)

Row spacing: its consistency among rows and its shape are important. Row spacing varies in different growing areas. Beet growers use a spacing of between 36 to 76 cm (14 to 30 in.).

Agricultural fields contain buried weed seeds, many of which can survive for a long period. When weed seeds germinate, they start to absorb the soil's nutrients. Kochia, pigweed, and lambsquarters are common weeds in sugarbeet fields. For better control of weeds, a **herbicide** (weed killer) can be applied prior to planting. Herbicides also reduce soil erosion (wearing away) because less tillage is required to kill the weeds.

Applying a herbicide before the plants emerge (pre-plant or pre-emergence) is a good control measure for most weeds. In the United States, an effective herbicide called Upbeet is used to kill Kochia. Also in the United States, postemergence herbicides such as desmedipham, triflusulfuron, and clopyralid are used at about 1.2 kg/ha (0.5 kg/acre or 1.2 lb/acre) by nearly all U.S. sugarbeet growers (Dexter 2005).

Sugarbeet is best grown in **crop rotation**, whereby other crops (mainly wheat, barley, and peas) are planted between years of beet planting. Crop rotation helps in the following ways:

- Improves soil fertility
- Prevents soil erosion and nitrate leaching
- Improves the yield and quality of the sugarbeet
- Reduces diseases and insects; for example, most pests attracted to corn will not damage the next season's crop of sugarbeet.

Most often, sugarbeet is grown with other crops in a three-year to five-year rotation. (*The longer the rotation, the better is the sugarbeet production.*) In Europe, 70 to 90% of sugarbeet is grown after cereal crops. In England, 10% of the farming area is sugarbeet and about 60% is cereal, so growing sugarbeet followed by wheat is common. In France, the fields are used four times with other crops between two sugarbeet crops (IIRB 2005).

Soil erosion from heavy rainfall causes the loss of good topsoil and can pollute nearby water sources. Ditches caused by erosion can create difficulties in prepared soil for planting. Polyacrylamides (its trade name in the United States is Soil Pam) precipitate soil particles and prevent erosion. This product is applied on the field at 1.2 kg/ha (1 lb/acre).

Soil pH is important to sugarbeet growth. Soil pH expresses the acidity or basicity (alkalinity) of the soil. The pH value is a dimensionless number (data with no unit) expressed on a scale of 1 to 14, with 7 as the neutral value. Acids have a pH from 1 to 7 (*the higher the pH, the weaker the acid*). Bases (alkalis) have a pH from 7 to 14 (*the higher the pH, the stronger the base*).

Sugarbeet grows best in a soil pH of 6.5 and higher. Hydrogen and aluminum ions in the soil are the primary source of soil acidity. Fertilizer containing ammonium salts (e.g., urea, ammonium nitrate, and ammonium sulfate) releases hydrogen ions during nitrification (conversion of ammonium salts to nitrate nitrogen, NO_3^-), causing the lowering of soil's pH.

The amount of carbon dioxide (CO_2) largely regulates the soil's pH. Carbon dioxide is formed by plant respiration, and reacts with water to form carbonic acid (H_2CO_3):

$$CO_2 + H_2O \rightarrow H_2CO_3$$

Therefore, the greater the amount of CO_2 in the soil, the more acid is formed and the lower is the pH of the soil. To raise the pH of the soil, calcium in the form of lime is applied. Some sources of calcium are calcite limestone, dolomitic limestone, quick lime, and hydrated lime. If magnesium also needs to be applied to the soil, it is best to use the dolomite form of lime.

Carbonation-lime residue (a by-product of beet-sugar factories) is a good pH enhancer at a reasonable cost. Lime residue (in Europe, it is called **Carbokalk**) ensures that soil contains sufficient lime. It improves aeration and drainage capabilities of the soil. Lime also reduces the risk of leaching. For liming, a typical requirement is about 5 t/ha (2 short tons per acre).

SUGARBEET PLANTING

Sugarbeet seeds are usually planted from late winter to early spring, depending on the weather conditions. In areas with mild winters, such as middle California, some beets are planted in the fall. The planting time is important. Because sugarbeet is a biennial plant, it does not get ripe and stop growing in the fall. Early planting gives the beets more time to grow.

Seed selection is important. In each sugarbeet-growing country, seed suppliers usually offer the best varieties suitable for the area. Today, beet growers are interested in seed varieties which are resistant to diseases and pests and produce a high-sugar content beet as well.

Seeds contain **starch** (a less soluble polysaccharide), which holds chemical energy. The energy is used for germination of the seeds. **Germination** (begin to vegetate) is complete in about a week at the normal temperatures of early spring (8°C or above). Adequate moisture and minimum soil compaction are two concerns at planting time and during seed germination and emergence.

The seeds are planted in the soil 2 to 4 cm (0.8 to 1.6 in.) deep, depending on the type of soil, the typical amount of rain or irrigation during germination, and the soil's temperature. **Planting depth** is important because shallow planting can cause the seeds to not grow if the soil is too dry, and planting too deep makes it more difficult for small plants to emerge.

Today, **mechanical planters** are used to plant seeds at different spacing. Seed spacing is usually from 7 to 15 cm (3 to 6 in.), depending on the row width. For planting, 12-, 16-, or 24-row **planters** are common. Figure 2.3 shows a 12-row planter placing the seed in the soil.

Planters also place dry fertilizer below and to the side of the seed. The planter's seed-spacing accuracy can be optimized by using an electronic test stand (Smith 2005). In general, a correct seed population and uniform seed spacing are required for a high sugarbeet yield.

Postplanting care starts once the crop begins to grow. It varies from area to area, as well as from field to field. Soil **cultivation** (loosening the soil surface), thinning, herbicide application, and fertilizing are the most important postplanting cares.

Soil cultivation loosens the soil surface, adds oxygen to the root area, and provides drainage and weed free area to apply herbicides. In mechanical cultivation, special care must be undertaken to minimize the damage to young plants.



Figure 2.3 A 12-row planter

Thinning is the removal of all superfluous plants, leaving only one plant in a spot as soon as the first leaves appear. With the development of monogerm seeds, thinning is an obsolete process. Monogerm seed has only one germ per seed, eliminating the thinning process.

Severe frost or freeze after the beet emerges is another concern. This can kill the young plant because sugarbeet is vulnerable during its early growth. Although the sugarbeet is a cold-tolerant plant, unusually cold temperatures in late spring can damage the young sugarbeet plant. In the case of severe damage, replanting may be required.

In the first six to eight weeks, the plant is vulnerable, but after the root is established, the plant is hardy (see Figure 2.4). Sugarbeet is a deep-rooted plant, with the root making up approximately 80% of the length of the plant. Normally, the hair roots are in the top 12 to 18 cm (5 to 7 in.) of soil, but the taproot can reach down to 2 m (6 ft) in the soil. Root hairs absorb nutrients and moisture from the soil. The total length of all root hairs from a single plant is estimated to be a few kilometers. Researchers at the USDA-ARS Michigan in the United States developed smooth root lines with less root hair. This type of beet holds less soil after harvesting, which is an advantage for the sugar factory because less water is required to clean beets and less soil (tare) must be hauled out. A three-year investigation of the Michigan fields has indicated that **soil tare** (soil sticking to sugarbeet) was reduced by 56.6% in using the smooth root lines compared to traditional varieties (Hubbell 2005). The results of this investigation have not yet been incorporated in commercial varieties available to growers.

The months of June, July, and August are important to plant growth. If full leaf canopy can be reached by the end of June, the maximum heat of sunlight is available to the plant, resulting in better growth. To mature for harvesting, sugarbeet needs 180 to 200 days, and approximately 1400 hours of sun. The average optimum temperature for growing sugarbeet during the vegetation period (from April through September) is about 14°C (57°F).

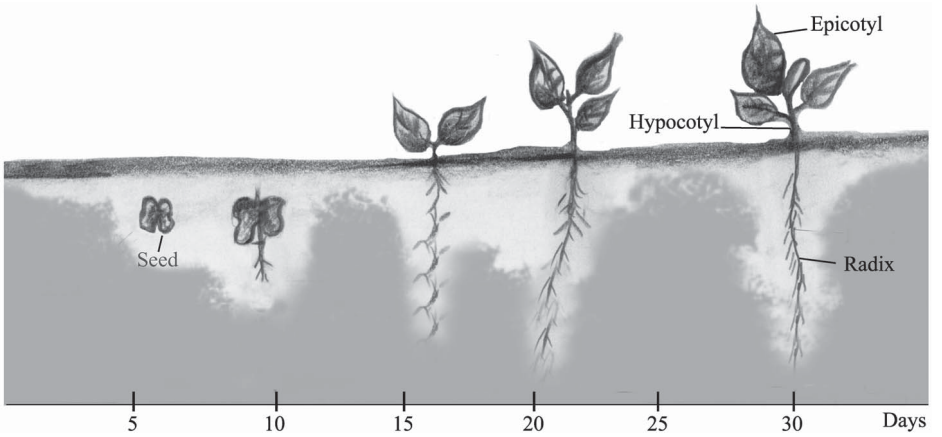


Figure 2.4 Early growth profile of sugarbeet

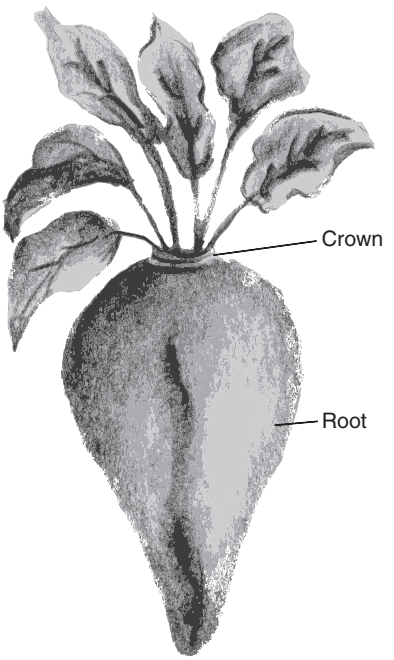


Figure 2.5 Grown sugarbeet plant

The sugarbeet root consists of the following three parts (see Figure 2.5):

- **Crown** (head or epicotyl): Grows above the ground and contains the leaves. Its height measures 2 to 3 cm (about 1 in.), depending on growing conditions.
- **Neck** (hypocotyl): Grows above the ground. Leaves grow from the hypocotyl. Its height measures 3 to 8 cm (1 to 3 in.), depending on growing conditions.

- **Root** (radix): Grows underground and is the main part of the root. Its typical length is 20 to 25 cm (8 to 10 in.).

A beet crown can have up to 30 leaves, with the old ones dying gradually. The shape of the leaf is generally oval to triangular, and the surface of the leaf is irregular and rough. The largest leaves can reach 45 cm (18 in.) or more in length. About half that length is **petiole**, a portion of the stem that supports the leaf. The rest is **leaf blade** (the flattened portion of the leaf).

Healthy sugarbeet leaves are important not only in the growth of the sugarbeet but also in its sucrose content. The sucrose is first synthesized in the leaves, and then moved to the **root cells** (Parenchyma cells) through the **phloem** (transport system of plant). The root (radix) is the usable portion of the sugarbeet, commonly known as the **sugarbeet**. Almost all sucrose is stored in the sugarbeet root (see Figure 2.6).

SUGARBEET FERTILIZING

Fertilizing is the addition of chemicals to soil to improve the soil's fertility. Fertilizing is one of the most important aspects of today's farming because the soil's fertility provides the nutrients that plants need. The three important elements for plants' growth are **nitrogen** (N), **phosphorous** (P), and **potassium** (K). The two groups of fertilizers are

- **Natural fertilizer** (compost): Produced from animal manure and dead plant materials such as sugarbeet top petiole and leaves, which are separated from the root during harvesting. Compost is the best soil supplement, but it is limited in supply.

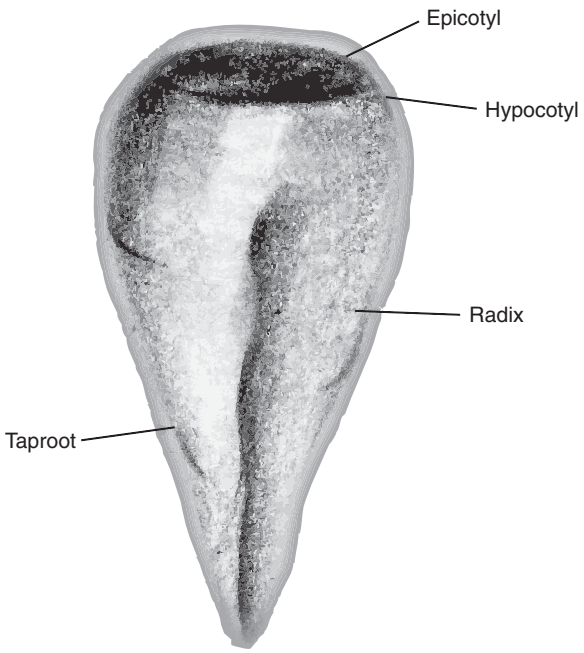


Figure 2.6 Sugarbeet root

- **Synthetic fertilizer** (industrial fertilizer): Produced from different chemicals and widely used. Synthetic fertilizer is needed to supply the world's population with food.

Synthetic fertilizers are grouped into two types:

- **Straight fertilizers:** Contain only one nutrient.
- **Mixed fertilizers:** Usually contain at least the three most essential nutrients, namely nitrogen (N), phosphorous (P), and potassium (K). Mixed fertilizers are labeled using the **N-P-K** system, which indicates the percentage of each element (the simplest types of matter, e.g., nitrogen). For example, 6-12-12 means that the fertilizer contains 6% N, 12% P, and 12% K.

The soil supplies many elements that are important to growing plants. Determining the correct fertilizer is difficult because fertilizers should contain several nutrients. Nutrients needed for growing plants can be divided into two groups:

- **Macronutrients**: Elements that plants need in large quantities. Nitrogen (N), phosphorous (P), potassium (K), calcium (Ca), magnesium (Mg), and sulfur (S) are the main macronutrients.
- **Micronutrients**: Elements that plants need in trace quantities. Iron (Fe), chlorine (Cl), boron (B), manganese (Mn), zinc (Zn), copper (Cu), and molybdenum (Mo) are the essential micronutrients.

Nitrogen (N) is the most important nutrient in the growth and quality of the sugarbeet plant. Production of most nitrogenous fertilizers starts with the reaction between nitrogen and hydrogen to give ammonia:

$$N_2 + 3H_2 \rightarrow 2NH_3$$

To grow fast, sugarbeet needs nitrogen, particularly early in the season. When the leaves form a canopy, the plant captures the maximum amount of sunlight. Extensive leaf growth beyond this is a waste. The highest quality sugarbeet (high-sugar and low-nonsugar content) is produced when nitrogen is used before the harvest. Nitrogen is needed to build leaves, which contain **chlorophyll** (the green pigment molecules responsible for photosynthesis). **Photosynthesis** is a complex reaction in plants by which carbon dioxide (CO_2) and water (H_2O) react together to produce saccharides (sugars) by absorbing energy from sunlight.

Naturally, plants get their nitrogen needs from soil and through nitrogen fixation. **Nitrogen fixation** is the process of absorbing nitrogen from the air (79% of air is nitrogen), converting it, and depositing it in the soil, where it can be used by the plant. Nitrogen fixation is performed by **bacteria** living in the soil. Dead plants are also a source of nitrogen because bacteria decompose the plants, releasing nitrogen. Plants use nitrogen in the form of **ammonium ions** (NH_4^+) and **nitrate ions** (NO_3^-). These ions are formed according to the following equations:

$$N_2 + 4H_2 \rightarrow 2NH_4^+$$

$$N_2 + 3O_2 \rightarrow 2NO_3^-$$

Plants can use ammonia (NH_3), nitrate (NO_3^-), urea (H_2NCONH_2), and other simple water-soluble compounds as their source of nitrogen. Plants are able to absorb ammonium ions (NH_4^+)

better than nitrate ions (NO_3^-) because **soil is negatively charged** and can absorb positively charged ammonium ions better than nitrate ions which are negatively charged. This property makes fertilizers with ammonium nitrate (NH_4NO_3) the most used nitrogen supplements.

The amount of the nitrogen (N) fertilizer depends on several factors, including the type of soil and the quantity of the residue of the crop grown in the field in the years prior growing sugarbeet. Nitrogen recommendations range from 100 to 165 kg/ha (90 to 150 lb/acre). Nitrogen can come from natural or synthetic fertilizer. Applying higher rates can reduce the quantity of the beets produced. Common forms of nitrogen to apply are anhydrous ammonia (82% N), urea (46% N), ammonium nitrate (34% N), ammonium sulfate (21% N), UAN solution (28% N), and mixed fertilizers. The nitrogen fertilizer can be supplied to the sugarbeet crop in one or a combination of the following ways:

- Apply before planting and mix it into the soil
- Place below and to the side of the seed while planting
- Put it into the soil between the rows while the beets are growing

Farmers use inorganic nitrogen fertilizer, **compost**, and **cattle manure** to supplement the nitrogen in the soil. Compost usually contains about 2% total nitrogen, of which one-fourth is estimated to be available to the soil. Compost use can increase sugarbeet yield but not increase the extractable sugar.

Nitrogen deficiency is usually recognized by *yellow leaves*. Older leaves look more yellowish than younger ones because nitrogen is mobile in the plant and moves to the younger leaves. Excess use of nitrogenous fertilizers should be avoided, particularly at the end of sugarbeet growing, because it slows sugarbeet growth and lowers its sucrose content and purity.

Phosphorous (P) is the second most important element in sugarbeet growing. Generally, phosphorous is used in plants for producing **nucleic acid** (a polymeric compound carried by every cell and involved in the transformation of genetic heritage) and energy-storing compounds, such as adenosine triphosphate (**ATP**). ATP is called the **powerhouse** of the plant because it is used to store energy in the cells. Phosphorous is particularly important for sugarbeet growth during the first period after planting. Both the quantity and the quality of the sugarbeet can be considerably improved by the right amount of phosphorous fertilizer.

Sugarbeet grown without an adequate amount of phosphorous develops **black-heart disease** (the foliage turns brown or black and the root decays). Plants can use phosphorous in the form of **phosphate ions** (PO_4^{3-}). Eroded phosphate-containing rocks and dead plants are the main natural sources of phosphates. Phosphorus is usually applied as P_2O_5 in a mixed fertilizer at planting time.

Potassium (K) activates enzymes used in photosynthetic process. The main natural sources of K are eroded rock, recycled dead plants, and potassium-containing fertilizers.

Symptoms for potassium deficiency in sugarbeet are the following:

- Yellowing of the leaves (usually along the edges)
- Reduction in petiole length

Sugarbeet grown with a low supply of potassium will have a lower sucrose content because of the reduction in photosynthesis. Potassium is usually applied as potassium carbonate known

as **potash** before planting or as a mixed fertilizer (N-P-K fertilizer) while planting. For sugarbeet, potassium is often applied at both times.

Minerals such as calcium and magnesium improve the sugarbeet growth rate and increase its resistance to pests and diseases. Minerals also bind to positively charged parts of the soil, preventing them from being washed away.

Plants absorb **calcium** (Ca) from the soil. Calcium ions (Ca^{2+}) are used for building cell walls. One of the best sources of calcium supplement for soil is **carbonation lime residue** (a by-product of the beet-sugar factory), which consists mainly of calcium carbonate (typically 80%), minerals (about 6%), and nitrogenous compounds (about 6%). Carbonation lime is left over from sugarbeet juice purification. Carbonation lime also neutralizes the acidity of the soil. Magnesium (Mg) is important for the formation of chlorophyll and for the activation of enzymes.

SUGARBEET GROWTH AND QUALITY

Sugarbeet growth depends mainly on the genetic of the variety, weather conditions, and the fertilizing program. Here, quality refers to sugarbeet purity, with high **purity** (percent sucrose in total dry substance) indicating high **sucrose** (sugar) and low **nonsucroses** (nonsugars). In climates where the growing season is longer due to weather conditions, the sugar content and yield are higher. High **sugar yield per acre planted** (known as the amount of **extractable sugar per acre**) is the most important factor in sugarbeet farming. Figure 2.7 shows the early growth of a sugarbeet field.

Cooperation between sugarbeet growers and processors is critical in the production of quality sugarbeet. From sugarbeet grower's and processor's points of view, quality sugarbeets have the following properties:

- High yield
- High sugar content



Figure 2.7 Sugarbeet plants during early growth

- Less nonsugar content
- Resistance to diseases
- Resistance to damage during transport
- Fewer root hairs to hold soil tare during harvesting
- Less harmful nonsugars to be processed efficiently in the factory

Experience indicates that cooperative sugar companies (known as co-ops) have been more successful than privately owned companies in producing quality sugarbeet. This is because growers are in charge of both sugarbeet production and sugarbeet processing. (For more detail on co-ops, refer to the Economy of Sugar subsection of Chapter 1.)

Sucrose (sugar) is a product of the **photosynthetic process**. Photosynthesis involves a series of complex reactions by which, in the sugarbeet plant, carbon dioxide (CO_2) and water (H_2O) react together to produce glucose and fructose (these two are monosaccharides). As the sugarbeet plant absorbs sunlight (solar) energy, the photosynthetic process occurs by two reactions: light and dark reactions. In the light reaction, chlorophyll absorbs light energy, producing monosaccharides, which then accumulates in plant molecules. In the dark reaction, the energy is used for the reaction of carbon dioxide with water to produce monosaccharides:

$$6CO_2 + 6H_2O + \text{Sunlight energy} \rightarrow C_6H_{12}O_6 + 6O_2$$

Monosaccharides are in the saccharides (the scientific name for sugars) family, which are a subgroup of the carbohydrate family (see the Carbohydrates section of Chapter 1). As glucose and fructose are formed by the photosynthetic process, solar energy becomes stored in the plant as chemical energy. The energy is used for further chemical reactions that promote plant growth. The combination of glucose and fructose to form sucrose with the help of enzymes is the most important chemical reaction taking place during sugarbeet growth:

$$C_6H_{12}O_6 + C_6H_{12}O_6 + \text{Enzyme} \rightarrow C_{12}H_{22}O_{11} + H_2O$$

(Note: Glucose and fructose are **isomers**, meaning they are compounds with the same *chemical formula* but different structures.) The chemical reactions occur in the leaves and move gradually with the flow of water to the root of the plant. Most of the monosaccharides, which are not converted to sucrose, stay in the leaves. Therefore, the amount of monosaccharides in the leaves is higher than in the root (1 to 3% compared to about 0.1% in the root).

For photosynthesis, plants absorb carbon dioxide from the air, water and minerals from the soil, and energy from the sun. For the reaction just shown to occur, the bonds between the carbon atoms of the carbon dioxide molecules and between the oxygen and hydrogen atoms of the water molecules must be broken. The atoms then recombine to form **glucose** ($C_6H_{12}O_6$) and **oxygen molecules** (O_2). Remember that breaking bonds always require energy, and bond formation releases energy. Therefore, the photosynthetic process is an **endothermic reaction** (an energy-requiring reaction). The energy required to drive this reaction comes from the sun.

Several enzymes are involved in the photosynthetic process. The overall rate of the process increases with air temperature and CO_2 content. In addition, the photosynthetic process cannot occur without the presence of leaves and their **chlorophyll** (the green pigment molecules in plant leaves that absorbs light energy). In the photosynthetic process, the chlorophyll interacts with **photons** (tiny packets of light energy) of sunlight and uses their energy. So, healthy leaves are essential in sugar formation. Sugar formed in the leaves is then moved to the root for storage.

The reverse of the photosynthetic reaction is the **respiration reaction**, by which sucrose and other carbohydrates are converted to carbon dioxide and water to produce the energy required for the beet's growth. During growth, the plant respires, and sucrose is decomposed to produce energy for growth:

$$C_{12}H_{22}O_{11} + H_2O \rightarrow C_6H_{12}O_6 + C_6H_{12}O_6$$

$$C_6H_{12}O_6 + 6O_2 \rightarrow 6CO_2 + 6H_2O + 2800\,kJ\ (669\ kcal)\ Energy$$

This reaction is an **exothermic reaction** (an energy-releasing reaction). Photosynthesis and respiration reactions are essentially opposite in terms of energy. Investigations indicate that under normal growing conditions, up to 40% of sucrose stored in the beet cell (the smallest unit that lives) is used for respiration and building the cell wall.

The basic unit of the sugarbeet (as with other plants) is the **cell** (see Figure 2.8). Because the sugarbeet cell is very small, it is not visible to the naked eye. Under a strong microscope, the cell diameter is observed to be about 50 micrometers ($1\,m = 10^6\,\mu m$); there are about 16000 cells in every mm^3 of sugarbeet root. A beet cell consists of

- **Cell wall:** Protects the cell. Consists mainly of cellulose and pectin
- **Protoplasm** (cell membrane): Controls the movement of molecules in and out of the vacuole. Consists of protein and is nonpermeable to sucrose and **nonsucrose substances** (all water-soluble substances except sucrose), but semipermeable to water.
- **Vacuole** (cell nucleus): Stores sucrose and nonsucrose substances. The vacuole is enclosed by protoplasm.

Besides weather conditions, sugarbeet growth depends on the following five factors:

- Water
- Oxygen

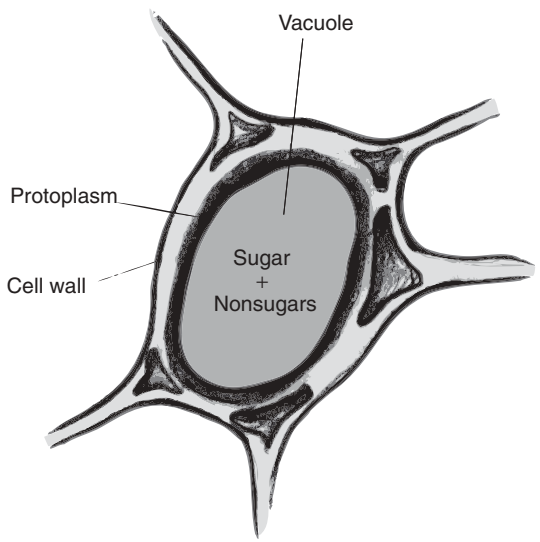


Figure 2.8 Beet-cell structure

- Carbon dioxide
- Organic compounds
- Inorganic compounds

The soil's **water content** is important because sugarbeet grows only in moist soil. On the other hand, excess water damages sugarbeet when the soil is saturated with water, which lessens the availability of oxygen to the plant.

The composition of the sugarbeet root is 75% water and 25% dry substance. The dry substance can be broken down into sucrose (about 17.5%), nonsucrose substances (about 2.5%), and about 5% **beet marc** (beet pulp). For more detail on sugarbeet composition, refer to Chapter 3.

The distribution of sucrose in the sugarbeet root is not uniform (see Figure 2.9). The most sugar gathers in the middle of the root, and the sugar content decreases rapidly toward the crown but slowly toward the tip of the root. The decrease in sucrose content is always accompanied by an increase in nonsucrose substances and by reduction in juice purity. Therefore, the amount of nonsucrose substances is higher in the areas that contain less sucrose.

SUGARBEET HARVESTING

Sugarbeet harvesting begins in mid-August and lasts until mid-September. The timing of the harvest is important; it is determined by sugarbeet ripeness, average root size, and weather conditions. Before harvesting, the mass of the root must be almost twice that of the crown, and the leaves start to turn yellow. With respect to root size, *the old belief that large roots contain less sucrose is not always true*. Harvesting time is the function of the following factors:

- Air temperature
- Length of sugarbeet storage

After the air temperature has cooled, beets can be harvested and put into pile storage. The optimum weather condition for harvesting is when the temperature drops to below 15°C (55°F). If the weather temperature is above 15°C, harvesting stops until the weather cools down.

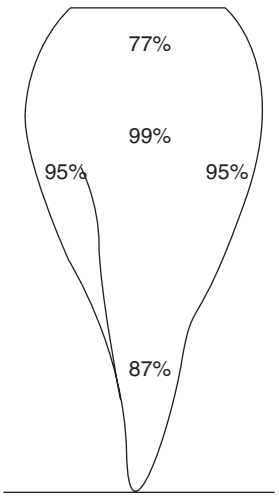


Figure 2.9 Sucrose distribution throughout the root

The planning of harvesting, transporting, and processing of beets in the factory is not easy to do. To allow a longer processing period, harvesting and processing normally start about one month before the temperature is cool enough to make piles to leave for extended periods. Until the temperature falls below 15°C (55°F), only a few days' supply of beets is required.

The length of sugarbeet storage is also a deciding factor in harvesting time. Long-term storage allows factories to slice beets for 150 to 250 days. In places where temperatures do not permit long-term storage (the warmer areas of Europe and North America, coastal areas of the Mediterranean Sea, and the Middle East), the harvest is conducted on an *as-needed basis*. In all circumstances, harvesting has to be completed before the first frost. Figure 2.10 shows the loading of beets onto a truck.

Sugarbeet is harvested (dug) out of the ground by a harvester. Two types of **harvesters** are used: **tractor-drawn** and **self-propelled**. Tractor-drawn harvesters remove the roots from the soil and remove some soil from the roots. The petiole (a portion of the stem that supports the leaf) and leaves are removed by a separate piece of equipment. Self-propelled harvesters remove the petioles and leaves in addition to removing the beet from the soil. Most harvesters lift beets from four to eight rows.

Topping is the removal of beet tops (petiole and leaves). **Scalping** is the cutting of the crown that follows the topping process. The amount of crown removed varies in different areas. In general, the beets are cut uniformly below the green leaf stalks of the crown (see Figure 2.11). Cutting the beets at the right level leaves a cut surface area of about 5 cm (2 in.) in diameter. If beets are cut above the normal level, the beet tonnage increases but so does the amount of nonsugars, which makes processing at the factory difficult. This also brings down the average sugar content of the beets, causing a decrease in factory efficiency and sugar production. In general, sugar losses during storage increase when beets are cut below or above the normal level.



Figure 2.10 A truck loaded with beets by a harvester

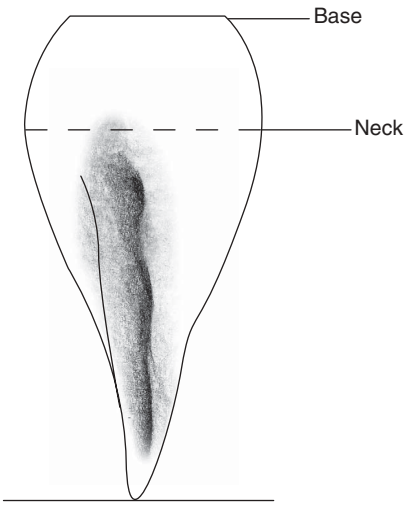


Figure 2.11 Topped beet



Figure 2.12 Defoliator

Defoliators remove the tops from the beets immediately after harvesting. This is accomplished by two or three sets of flails, of which usually one or two sets are made of rubber, but one set is made of steel (see Figure 2.12).

When the roots are harvested, sugar accumulation in the sugarbeet stops. However, during **storage**, the *tharvested beet* continues to live by **respiration** and consumption of its sugar, which is considered **sugar loss**.

During harvesting, some soil remains attached to the root along with some sand and stone. Impurities (clay, sand, stone, and trash mixed with the beets) carried with the beets to

the factory are called the **beet tare** (or just **tare**). The amount of beet tare and its reduction during harvesting are important to the sugar factory. The soil tare creates problems in storage as well as disposal issues after the soil is removed from the roots at the factory. Modern harvesting uses cleaner equipment and better storage area to reduce soil tare. Beet tare is measured at the time of beet delivery by the tare lab operators.

The beet storage method and the amount of tare can contribute to **sugar loss** during storage. Soil, weeds, and leaves that are piled with the roots reduce air movement during cooling and can increase sugar loss in storage.

Beets are usually transported by large trucks or by railcars (depending on the proximity to the railroad), to be stockpiled in the **beet-storage areas** (beet piling grounds) in piles 5 to 12 m (15 to 36 ft) high. Beets are usually piled by mobile pilers.

A **beet piler** (see Figure 2.13) consists of a receiving hopper, a belt conveyor, a screen to remove soil and trashes, and a large boom (up to 30 m) to put the beets in a pile. Some pilers are also equipped with a sampling device. A large piler can pile up to about 500 t of beets per hour. As the beets are passed over the screen, any soil that is screened out is put back in the truck. The amount of removed soil is typically about 50% of what comes in with the beets, but this depends particularly on the speed of the piler, the size of the screen, and the moisture content of the soil (claylike soil is difficult to separate from beets).

Keeping beet piles under suitable conditions (particularly during long-term storage) is very important to the efficiency of a sugar factory. Therefore, pile management is an important responsibility of the agricultural department. The three types of beet storage are as follows:

- **Factory storage**: Located in the vicinity of the factory
- **Remote storage**: Located between the fields and the factory
- **Clamp storage**: Located in the farmer's field



Figure 2.13 Beet piler



Figure 2.14 Beet storage



Figure 2.15 Beet receiving and piling at night

Figure 2.14 shows a factory beet storage. For more information about beet storage, refer to Section 1 of Chapter 3.

As shown in Figures 2.15 and 2.16, during **harvesting**, beets are delivered and piled day and night. Because sugar loss occurs during storage, the factory must process the beets quickly. In winter-beet-growing areas with warm climates (e.g., North Africa, some parts of Asia,



Figure 2.16 Beet handling with a loader

southern Europe, and South America) where the average daily temperature is above 30°C (86°F), sugarbeets delivered to the factory must be processed within a week to prevent high sugar loss. Therefore, the harvesting is usually stretched out over a three- to five-month period, and growers deliver their beets according to a schedule. In northern Europe and North America, the harvesting time is much shorter (four to six weeks), and beets are stored up to six months because of the low temperatures.

SUGARBEET YIELD

In the beet farmer's language, **stand count** (or simply stand) indicates how many sugarbeet plants grow in the row. Stand is often counted as sugarbeets per 30 m (100 ft) of row. So, root population and weight are the major factors in stand count. Stand count is important, especially where irrigation is not available. In the United States, a good stand ranges from 140 to 170 beets/100 ft. However, stands of 60 to 100 can be acceptable, depending on the conditions.

In the United States, about 0.4 kg (about 1 lb) of seed is planted per acre of field. This results in a sugarbeet yield of 15 to 25 short tons per acre (about 6.6 to 11.0 t/ha), depending on the growing conditions (average 23+/acre in 2005). The average yield of 15 European countries is about 8.1 metric tons per hectare. For example, from an average of 20 tons per acre and a recovery of 277 lb of sugar per ton beet, 1 lb of seed will produce 5540 lb (2.8 short tons) of granulated-refined (GR) sugar.

Seed quality plays an important role in sugarbeet agriculture. For centuries, farmers have improved crops by breeding seed for desirable characteristics. Besides sugarbeet size, increase in sucrose content, decrease in nonsucrose content, and resistance to disease and pests are the aim of seed producers.

SUGARBEET SEED PRODUCTION

Sugarbeet seed is produced in areas with ideal agricultural and climatic conditions (e.g., the Willamette Valley in Oregon and some parts of France and Italy). Production-quality sugarbeet

seed is a delicate process. On average, it takes several years of experimenting and cross-examining several plants to develop a quality sugarbeet hybrid seed. Sugarbeet seed is produced by exclusive nurseries and some beet-sugar companies.

Today, only monogerm sugarbeet seed is used because of the unpopularity of multigerm seeds among growers. There are different methods of seed production for planting seed mixture with different amounts and types of pollinators (male components) and male-sterile plants (female components). Among other methods, hybrid sugarbeet seed is produced by capitalizing on cross pollination and on the sterile-male plant. Male and female components are planted in rows 0.5 to 1.0 m (1.7 to 3.3 ft) apart (much larger than seed spacing) in August or early September. Seeds go through germination, emergence, and pollination (on the female plants). Seeds are harvested around the next August. Harvested seeds are cleaned to separate impurities and processed before being sold to farmers. Seed processing is important to prevent contamination by foreign pollens. Seed processing consists of the following steps:

- Steeping (first wash) and priming (second wash)
- Drying to evaporate moisture and make seed harder
- Pelleting naked seeds by covering them with a wood flour
- Coating seeds with insecticide and fungicide to protect against seedling process

Sugarbeet seeds are ball-shaped and about 5 mm in diameter (see Figure 2.17). Seeds contain the nutrients (e.g., carbohydrates and proteins) that serve as food until the young plant emerges from the soil. Because the sugarbeet is a biennial plant, it takes two years to mature and produce seeds. Figure 2.18 shows a sugarbeet plant stalk with seeds on it.



Figure 2.17 Two types of Sugarbeet seeds

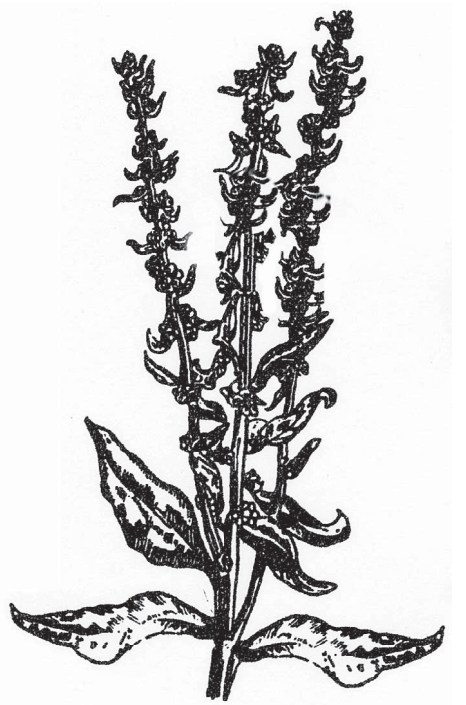


Figure 2.18 Sugarbeet plant with seeds

SUGARBEET DISEASES AND INSECTS

Damage to the sugarbeet leaves and roots can significantly lower the quality and yield of the plant.

The following are key factors that influence the rate of **sugarbeet disease**:

- Pests
- Insects
- Soil moisture
- Air temperature
- Soil temperature

The young sugarbeet plant is very vulnerable to diseases such as Rhizoctonia, Cercospora leafspot, Aphenmyces, and Pithium, as well as damage caused by insects. Cercospora beticola leafspot, Rhizoctonia solani, curly top, and Rhizomania are diseases that can damage plants in late summer. Some insects are beneficial to agriculture, but a small number of them threaten the plant's growth. (When using insecticides, pay close attention to preventing destruction of beneficial insects.)

Insects such as the leafhopper, cutworms, root lice, white grub, the flea beetle, the carrion beetle, and the root maggot carry viral diseases. For example, the leafhopper carries the curly-top virus, and the Piesma cinerea (plant bug) carries beet Savoy. Worldwide, one of the most

devastating sugarbeet diseases is caused by Cercospora beticola, commonly known as Cercospora Leafspot.

Rhizomania is a viral disease carried by a soil fungus, which is widespread throughout the world. Sugarbeets infected with Rhizomania have damaged roots, which create difficulties in storage and processing.

Curly top is a viral disease carried by the beet leafhopper. Sugarbeets infected with curly top show leaf curling, discoloration, and stunning; often young plants die.

Nematodes are round worms about 1 mm long. Many types of nematodes exist, the most common is the **beet nematodes** (*Heterodera schachtii*), which inflict serious sugarbeet damage. Plants resistant to nematodes, such as rye and corn, are introduced during the crop rotation to control these pests. Certain varieties of oil-seed radish can trap the nematode cyst. The cyst nematodes hatch but are unable to feed on the oil-seed radish plants' roots, so they die.

Beet weevils (see Figure 2.19), light- or dark-gray beetles 10 to 15 mm long, live underground in the winter in sugarbeet fields. In spring, they start to feed on sugarbeet leaves. Root aphids (*Pemphigus populivenae*) also feed on beet roots.

Sugarbeet plant diseases vary from area to area, and what works in one area may not necessarily work in another. Different seed varieties offer varying levels of resistance to sugarbeet diseases and pests. Choosing the right seed variety in sugarbeet production helps decrease the use of chemical pesticides.

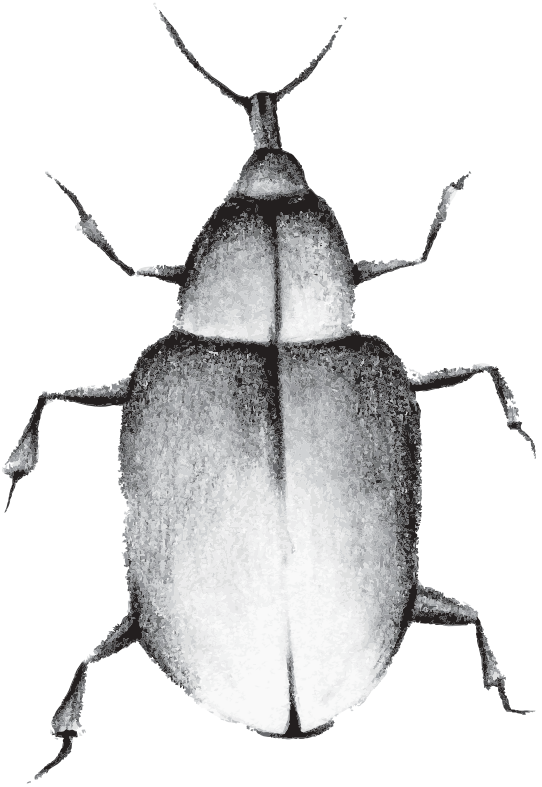


Figure 2.19 Beet weevil

Sugarbeet farmers use toxic chemicals to fight against diseases, weeds, and insects. In agriculture, the term **pesticide** is used to describe insecticides (destroy insects), fungicides (destroy fungus), herbicides (destroy weeds), insecticides (destroy insects), and nematicides (destroy nematodes). **Safety precautions** are required during the application of these chemicals because of their toxicity. For example, insecticides have the potential to kill honeybees, a beneficial insect.

Herbicides are used by farmers to kill weeds, which also consume the nutrients and water that sugarbeet needs. The traditional method for controlling weeds is to remove them by hand which is labor-intensive. Today, weeds are controlled mechanically and with herbicides.

Fungicides are used to control diseases caused by fungi. Moisture, high humidity, and high temperatures can lead to ***Cercospora beticoli*** infections in August and September. This fungus can cause significant loss to the plant if not controlled with fungicides. Sugarbeet varieties differ greatly in their resistance to various diseases. Many classes of fungicides are on the market. In the United States, Azoxystrobin (Amistar) is a commonly used fungicide for the control of Rhizoctonia solani diseases.

SUGARBEET AND GENETIC ENGINEERING

Genetic engineering (GE) is the **breeding** process in its scientific form. Thus, it can be used under governmental regulations (such as those given by USDA and FDA) to improve crops. Humans have used breeding since the prehistoric time as the process for producing new varieties of organism (plants or animals) with improved characteristics. Traditional breeding is a lengthy process and the results may be uncertain, while those of GE are fast and almost absolute.

GE is based on the recombination of the gene system of an organism to transfer the desired characteristics. The **gene** is a unit of heredity in the cell's chromosome, which consists of deoxyribonucleic acid (**DNA**). In a **genetically modified organism** (GMO), one (or more) DNA from an organism is combined with the DNA system of another organism. The organism with recombined DNA (rDNA) is a GMO. For example, if a microorganism produces a protein that is toxic to beet weevil, by inserting the microorganism's gene into seed DNA, the GM beet could produce the same protein in its cells and therefore become resistant to the beet weevil.

Farming based on genetic engineering is known as **transgenic agriculture**. Today, sugarbeet research institutes (22 just in Europe) are focusing large efforts in the area of GM crops, which were first introduced for commercial production in 1996. Since then, genetic scientists have been researching to improve the quality of sugarbeet, such as the following:

- Yield
- Sucrose content
- Selectivity of nonsugars
- Resistance against weather conditions
- Resistance against diseases and other pests

As in any new invention, the consumer's view is uncertain about how the modified crops are produced. Some consumers have concerns about the safety of GM foods. Although it is difficult to certify that there are no rDNA in a particular lot of beet (because of contamination by foreign pollens during seed production), some advanced countries in sugarbeet production have

not yet approved the cultivation of GM sugarbeet. In general, scientists believe that over time, GM crops will be accepted by a majority of consumers.

ORGANIC SUGARBEET

Organic farming is a term used to indicate farming done without the use of chemicals that are not synthesized but are produced **naturally**. Foods prepared without the use of synthetic fertilizers and pesticides, growth hormones, antibiotics, or genetic engineering are called **organic foods**, which are increasing in popularity.

Unlike the conventional farming (uses synthetic fertilizers and pesticides), organic farming uses **natural fertilizer** (compost) as the soil's supplement and crop rotation as protection against plant diseases. Besides ordinary compost (manure), organic farmers use compost–mineral blends and locally available fertilizing materials such as poultry manure, compost tea, pure and enhanced microbial cultures, ashes, and other available materials. In **composting** (the process of breaking down organic compounds by microorganisms, and then building that material into humus), the type of microorganisms used is important.

In organic farming, crop rotation works well because of different pests' habits to attack crops. **Biopesticides** are used in organic farming as pesticides. Biopesticides are derived from natural materials, plants, bacteria, and certain minerals. Baking soda and canola oil are two examples of biopesticides.

Organic farming is strictly controlled. In 2002, the United States Department of Agriculture (USDA) released the National Organic Standards, which allows farmers to label their products as Certified Organic if they contain *more than 75% organic ingredients*.

Organic sugarbeet is produced for production of organic sugar. Organic sugarbeet farming is a relatively new development, dating back only about 10 years. According to the International Sugar Organization (ISO), organic sugarbeet farming in the world is experiencing a growth rate of 20 to 30% annually, and it reached about 200000 tons in 2005. In Europe, the British Sugar Corporation started in 2001 to process organic sugarbeet with an initial production of 10000 t per year. Organic sugarbeets are processed at the beginning of the campaign, separate from normal beets. Organic sugar is used in organic ice cream, jam, baked food, and confectionary (Smith 2003).

The price of organic foods is now two to three times higher than ordinary food products. However, the prices are expected to come down as organic food production increases.

Internationally, the organic food industry has been growing rapidly. In the United States, it has grown more than 20% annually since 1990. The main reason for this high growth is a rising popular concern for human health and the natural environment.

SUGARBEET AND NATURAL ENVIRONMENT

Sugarbeet is an environmentally friendly crop that is planted in many countries worldwide. Compared to other crops in rotation with sugarbeet such as corn, sugarbeet farming provides the following environmental advantages:

- Lower nitrogen requirement
- Better wildlife population
- Better erosion control

- Better landscape
- Cleaner soil

During the summer and fall, the sugarbeet canopy provides shade for the soil surface below, creating up to a 20°C difference between the air and soil surface temperature in hot areas. This provides nesting sites for many ground-nesting birds such as the skylark. After the sugarbeet is harvested, the field is undisturbed for a few months, making it a good food source for birds. More than a quarter of the world's pink-footed geese feed on beet fields (IIRB 2005). Mammals can also feed on sugarbeet fields.

Soil water erosion during heavy rainfall, particularly in hilly areas, damages soil structure and washes nutrients away. Sugarbeet farming stabilizes the soil structure by covering the soil surface with vegetation as the beets become established. Sugarbeet also prevents soil erosion by wind. This is helpful in areas close to the sea and in flatlands.

NOTES

- **Agriculture in today's society must supply six billion people with food. By the end of this century, the earth's population will likely double. Because the food supply and the sugar supply must increase accordingly, the expansion of agriculture must take into account human health and the environment in terms of soil and air quality.**
- **For more information on sugarbeet and related environmental issues, refer to the comprehensive article published by the International Institute for Beet Research (IIRB) in *Zucker Industrie* of February 2005.**

BEET LABORATORY AND GROWERS' PAYMENT

The **beet laboratory** (some call it a **tare laboratory**) is a small laboratory (lab) usually set up close to the beet-receiving area. Each sugar factory has a main lab, which is in the main processing building (see Chapter 11 for more information about the laboratory and method of analyses). The main function of the beet lab is to determine **beet tare** (clay, sand, stone, and trash mixed with the beets) and **sugar** (sucrose) content. The data from the beet lab is then used for calculating the growers' payments.

The **growers' payment** for delivered beets is based on the **beet net weight** (weight of tare-free beets) and **sucrose** (sugar) content. **Beet-receiving stations** are equipped with a sampling device, which is installed in front of the weighing scale (usually beets are transported by truck or railcar).

The sampling, processing of the sample, and computing growers' payments are different in different countries (and sometimes from factory to factory within the same country). These methods are based on contracts between the sugar company and the sugarbeet growers. The following method is used in some U.S. beet-sugar factories.

1. A representative sample (usually 10 to 12 kg, or 20 to 25 lb) is taken from each grower delivery and sent to the beet lab.
2. The sample is weighed (W_1) and recorded in the computer.
3. Tare is determined by removing the trash and brushing (or washing) the beets to remove all nonbeet materials from the sample.
4. The clean beet sample is weighed again (W_2) and recorded in the computer.
5. A **beet brei** is produced from the sample by sawing beets with a machine equipped with a rotating saw blade (see the Notes).

6. The brei is placed onto reinforced paper and squeezed to remove the juice from the pulp.
7. The juice is poured into a plastic bottle and placed in a tray that holds 21 bottles.
8. The bottle is labeled with grower's contract number and kept in a freezer.
9. After 21 samples from the same grower are collected, the tray is taken from the freezer, and defrosted at room temperature.
10. The contents of all 21 bottles are mixed to prepare a composite sample.
11. The sample is tested for the percentage of sucrose (sugar) content, usually by the **cold digestion method** (see Sugar-in-Cossettes subsection in Section 4 of Chapter 11 for the analytical procedure).

Tare (clay, sand, stone, and trash mixed with the beets) is calculated according to the following equation:

$$\%\text{Tare} = \frac{W_1 - W_2}{W_1} \times 100 \tag{2.1}$$

The **beet gross weight** (weight of beets and beet tare) equals the weight of the loaded vehicle (**weight in**) minus the weight of the unloaded vehicle (**weight out**):

$$\text{Gross weight} = \text{Weight in} - \text{Weight out} \tag{2.2}$$

All tares for a particular grower for each day are averaged and applied to beet gross weight to calculate **beet net weight**:

$$\text{Net weight} = \frac{(\text{Gross weight})(100 - \%\text{Tare})}{100} \tag{2.3}$$

Payment factor is calculated according to the following equation:

$$\text{Payment factor} = \frac{\text{Average \%sugar each grower}}{\text{Average \%sugar all growers}} \tag{2.4}$$

Grower payment is determined by payment factor multiplied by base price of beet:

$$\text{Grower payment} = \text{Payment factor} \times \text{Base price} \tag{2.5}$$

EXAMPLE

Given:

W_1	12.52 kg
W_2	11.92 kg
Delivered beet gross weight	25.55 t

Calculate:

% Tare

Beet net weight

$$\text{Tare} = \frac{W_1 - W_2}{W_1} \times 100 = \frac{12.52 - 11.97}{12.52} \times 100 = 4.4\%$$

$$\text{Net weight} = \frac{(\text{Gross beets})(100 - \%\text{Tare})}{100} = \frac{25.55(100 - 4.4)}{100} = 24.4t$$

NOTES

- **To some extent, the analytical results of the beet laboratory depend on the brei preparation, so the method for sawing beets sample is usually written into the contract between the sugar company and the sugarbeet growers.**
- **For more information on beet tare and loading and unloading sugarbeets, refer to Section 1 of Chapter 3.**

SUGARBEET FARMING PROBLEMS

Growing sugarbeet is not easy. It requires sufficient theoretical knowledge and experience. Even experienced sugarbeet growers might face some problems while growing sugarbeet. The following are a few examples of those problems.

Sugarbeet with Low Yield

High sugarbeet yield with high sugar content is the goal of all sugarbeet growers. Possible causes of low yield (poor stands) are as follows:

- Late planting
- Wrong seed variety
- Insufficient weed control
- Insufficient fertilizer application
- Insufficient disease and insect control
- Insufficient irrigation in irrigated areas or low rainfall in nonirrigated areas

Sugarbeet with Low Sugar Content

Following are the possible causes of low sugar content in sugarbeet:

- Unexpected disease
- Improper seed variety
- Unusual weather conditions
- Excess nitrogen fertilizer application
- Poor removal of petioles and leaves during harvesting
- Insufficient growing period (sugarbeet needs about 150 days to mature)
- Sugarbeet damaged during harvesting by the use of the wrong equipment or improper operation

Sugarbeet with High Crown Content

Following are the causes of sugarbeet with high crown content:

- Excess nitrogen available during the late growing period
- High nitrogen fertilizer application

Sugarbeet with High Tare

Beets received at the factory will contain **tare** (impurities), which has to be separated from the beets during the early steps of the process. Soil, weeds, and leaves mixed with the beets in

storage piles cause higher sugar loss and spoilage. In addition, high tare increases processing costs in terms of damage to equipment, water consumption, and wastewater treatment. The amount of tare can range from 2 to more than 8% on beet (**OB**). In clay-type soil areas or if farmers harvest beets in rainy weather, the tare is higher. Due to different sugarbeet-growing conditions, determining the optimum level for tare content is difficult. However, the following are causes for high tare:

- Fine-textured soil
- Harvesting too quickly
- Insufficient weed control
- Wet conditions during harvest
- Sugarbeet grown in eroded fields
- Sugarbeet with rough surfaces (holds more soil)
- Sugarbeet with more root hairs (holds more soil)
- Insufficient removal of petioles and leaves during harvesting

Following are a few conversion factors that are helpful for converting agricultural metric units into English units, and vice versa:

Hectare = 10000 m^2
Acre = 4047 m^2 = 43500 ft^2
Hectare × 2.47 = acre
Acre × 0.405 = hectare
Metric ton × 1.12 = short ton
Short ton × 0.91 = metric ton
Short ton/acre × 2.27 = metric ton/ha
Metric ton/ha × 0.44 = short ton/acre
lb/acre × 1.12 = kg/ha
kg/ha × 0.89 = lb/acre

END-OF-THE-CHAPTER NOTES

- **Sugarbeet farmers cannot work well "by just the book", it needs a great deal of knowledge and experience. Fortunately, sugarbeet farmers are not alone in their efforts. Agriculturalists of the factory offer valuable recommendations to assist sugarbeet growers, inspect sugarbeet fields, maintain records of field productivity, assist in research, supervise delivery and storage of sugarbeet, and answer farmers' questions with regard to sugarbeet growing. They also help farmers in the selection of suitable seed, fertilizer, herbicide, and other materials. Relatively, there is more cooperation between sugarbeet farmers and processors than is found with other agricultural products.**
- **Timing is the most important element in sugarbeet growing. Planting, fertilizing, weed killing, and finally harvesting at the right time produce quality sugarbeet with high yield.**
- **In Chapter 2, you have learned how quality sugarbeet is grown in the field. Chapter 3 (Sugarbeet Processing) discusses the stations involved in sugar manufacturing and addresses problems that sugar technologists face in their routine work.**

CHAPTER

SUGARBEET PROCESSING 3

SECTIONS

1. Beet Receiving and Storage
2. Beet Drycleaning
3. Beet Conveying and Fluming
4. Stone and Trash Separation
5. Beet Washing and Flume-Water Treatment
6. Beet Slicing
7. Juice Diffusion
8. Pulp Treatment
9. Milk-of-Lime and Carbonation-Gas Production
10. Juice Purification
11. Sedimentation and Filtration
12. Steam and Power Production
13. Juice Evaporation
14. Juice Decolorization and Sulfitation
15. Juice Storage
16. Syrup Crystallization
17. Molasses Exhaustion
18. Massecuite Centrifuging
19. Sugar Drying, Storing, and Packing
20. Production of Specialty Sugars

ABOUT THE CHAPTER **Chapter 3** is a systematic study of the unit operations in sugarbeet processing. Sugar production depends on large-scale operations that consist of several unit operations. Successive unit operations are involved in separating **sugar** (sucrose) from **nonsugars** (nonsucroses). The nonsugars (impurities) are the undesirable substances.

Beet-Sugar Handbook, by Mosen Asadi

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Separation of sugar from nonsugars in fact is the aim of almost every step of sugar production. The sugar separation is gradual and is accomplished in several stations. The improvement in each station is expressed by the **purity** (sugar content as % of dry substance) of the product from that station. As Figure 3.1 shows, the ultimate goal is to produce sugar with almost 100% purity.

In sugarbeet processing, the term **station** is used to denote the section of the factory that does a particular job. To make sugarbeet processing easier to learn, this chapter is divided into 20 sections, each corresponding to a station of sugarbeet processing.

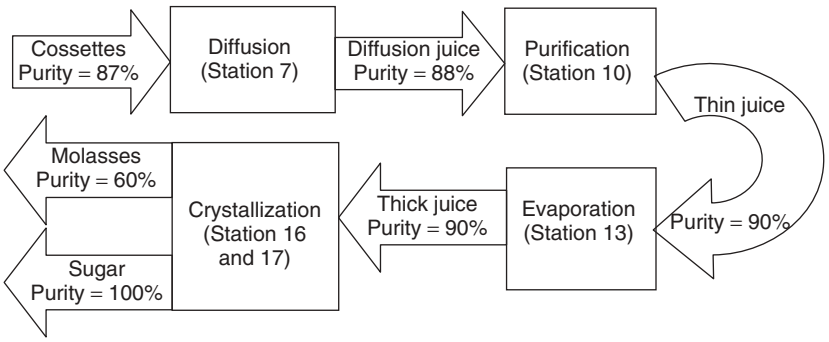


Figure 3.1 Purity profile

The **agricultural department** of the sugar factory is responsible for coordinating with sugarbeet farmers the delivery, unloading, and storing of sugarbeets, as well as care of sugarbeets during storage. At the point where the factory starts to process sugarbeets, responsibility passes to the **operation department**, which must recover as much sugar as possible from the sugarbeets entered into the process.

Changing sugarbeet to sugar requires successive processes, called **unit operations**. The unit operation is one of the subjects that differentiates chemical engineering from chemistry at universities. Unit operation studies focus on the chemical processes used in chemical and food factories. Sugar production is a large-scale operation divided into smaller unit operations. To process sugarbeet and produce sugar, 10 unit operations are commonly used in a beet-sugar factory:

- Fluid transportation
- Energy utilization
- Heat transfer

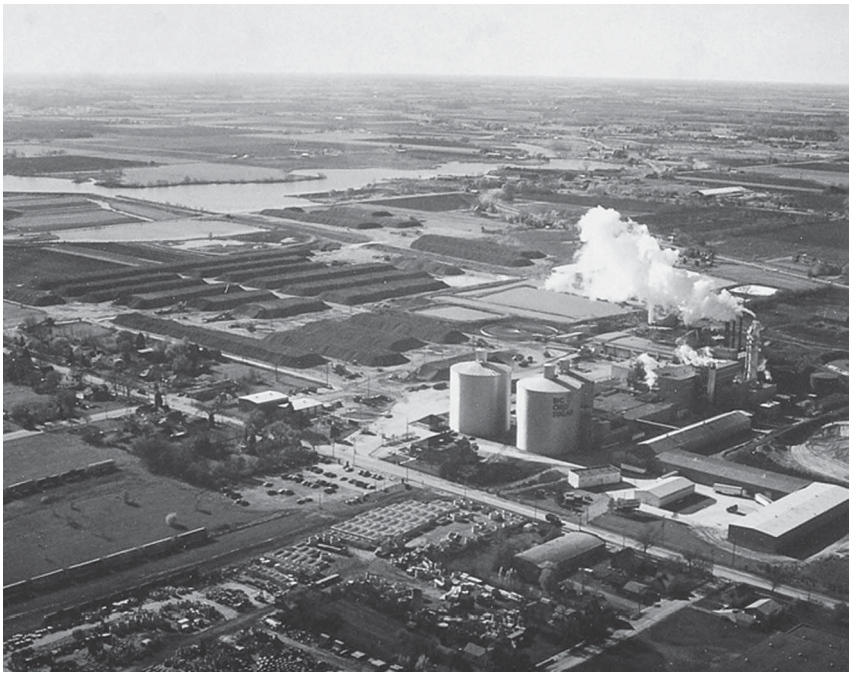


Figure 3.2 A beet-sugar factory (Courtesy, Michigan Sugar Company)

- Diffusion
- Filtration
- Sedimentation
- Evaporation
- Crystallization
- Centrifugation
- Drying

In addition, today, the beet-sugar industry uses **ion-exchange resin technology** in juice softening (Chapter 6), molasses desugaring (Chapter 8), and in some other processes. Furthermore, microfiltration has been under investigation by the beet-sugar industry for the last two decades as an attractive unit operation in the juice-purification process (see Section 10 of this chapter). Technologies based on resin are different from those used in conventional sugar operations. Condidering all the above, the process of sugar production is complex and requires operators with a good knowledge of sugar technology and a desire to learn new techniques.

To change raw material (sugarbeet) into sugar, the sugar factory has to separate nonsugars (all soluble substances except sugar) in the beet juice from sugar. Several stations are required to separate nonsugars (unwanted components) from sugar (wanted component). In this chapter, sugarbeet processing is subdivided into 20 stations, which are explained in a systematic order.

(Each station corresponds to a section of the factory assigned to do a particular job.) As Figure 3.3 shows, the first 13 stations (through the evaporation station) make up the beet-end of the factory; the remaining stations are in the sugar-end.

Each station consists of two or more steps, totaling 80 to 85 steps. For example, the purification station (the station with highest number of steps) consists of the following 13 steps: juice heating, preliming, prelimed-juice heating, mainliming, limed-juice heating, first carbonation, mud separation, mud thickening, first-carbonation filtration, first-carbonation juice heating, second carbonation, second-carbonation filtration, and second-carbonation safety filtration.

Figure 3.4 shows a simplified flow diagram of a typical beet-sugar factory (the numbers in parentheses indicate the stations). In this figure, **in-process products** (juices, syrups, liquors, and massecuites) go through the factory along the solid line. By-products (pulp, carbonation-lime residue, and molasses) and other materials (e.g., limestone, coke, and steam used in processing) are shown by a broken line.

NOTES

- **Some factories do not have all these stations. For example, some factories do not have the beet-drycleaning, juice-decolorization, or juice-storage stations.**
- **In recent years, use of computerized control systems for automation and control of different equipment in sugar factories has greatly increased. These systems are rapidly replacing the traditional electromechanical control systems.**
- **Today, not only does availability of improved automation systems help control equipment, it also helps sugar factories install equipment in out-the-main-building arrangements in open areas. Diffusers (especially, the tower diffusers), prelimer, carbonation tanks, and continuous crystallizer pans have been recently installed in this way. Where the climate is mild, even steam-pulp dryers, evaporators, filters, first-carb clarifiers, and more beet-end equipment can be installed outside. All equipment so installed can be controlled from the main control room of the factory (see Figure 3.5).**

Beet-end		Sugar-end	
1	Beet receiving and storage	14	Juice decolorization and sulfitation
2	Beet drycleaning*	15	Juice storage*
3	Beet conveying and fluming	16	Crystallization
4	Stone and trash separation	17	Molasses exhaustion
5	Beet washing and flume-water treatment	18	Centrifuging
6	Beet slicing	19	Sugar Drying, Storing, and Packing
7	Diffusion process	20	Production of Specialty Sugars
8	Pulp treatment		
9	Milk of lime and carbonation gas production		
10	Juice purification		
11	Juice sedimentation and filtration		
12	Steam and power production		
13	Juice evaporation		

* Some factoried do not have these stations

Figure 3.3 Stations of the beet-sugar factory

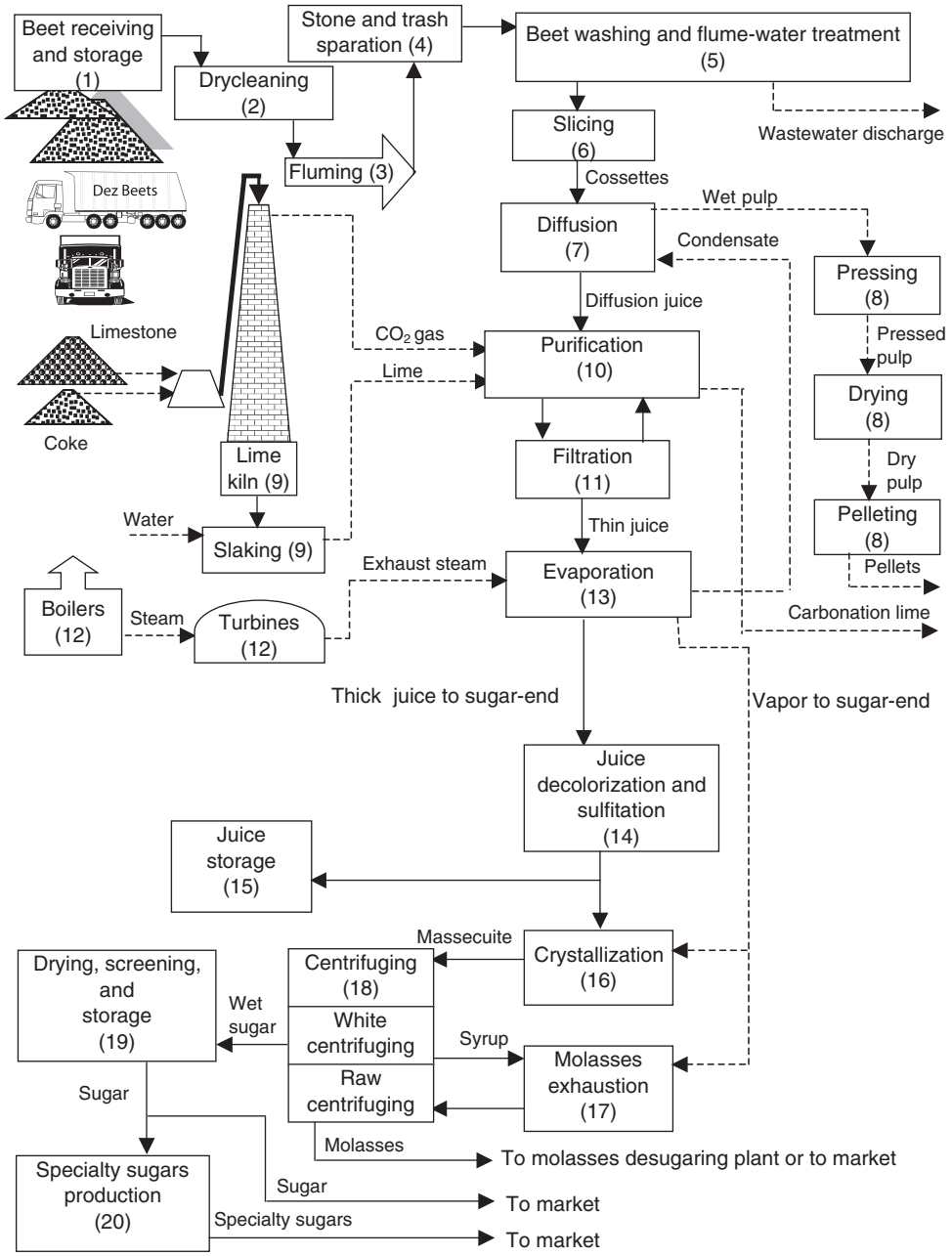


Figure 3.4 Flow diagram of a typical beet-sugar factory



Figure 3.5 Sugar factory's control room

SUGARBEET COMPOSITION

Sugarbeet (the raw material of the beet-sugar factory) **composition** is important to both the sugarbeet farmer and the processor (factory). Today, sugarbeet is purchased by quantity and quality. **Sugar** (sucrose) and **nonsugar** (nonsucrose) content determine the quality of the sugarbeet (high sugar and low nonsugar content is desirable).

The basic unit of sugarbeet (like other plants) is the **cell**. A beet cell consists of

- **Cell wall:** Protects the cell and consists mainly of cellulose and pectin.
- **Protoplasm** (cell membrane): Controls the movement of molecules in and out of the vacuole. The protoplasm consists of protein and is nonpermeable to sucrose and nonsucrose substances but semipermeable to water.
- **Vacuole** (cell nucleus): Stores beet juice, containing sucrose and nonsucrose substances.

As shown in Figure 3.6, the dry substance of sugarbeet consists of beet juice and beet marc (beet pulp). Beet juice contains both sucrose and nonsucrose (impurities).

Sugarbeet juice is mainly made up of **saccharides** (sugars), which are sucrose (15 to 20%), raffinose (0.2 to 0.5%), glucose and fructose (0.05 to 0.1%), and planteose, stachyose, and verbascose (in trace amounts). Beet juice always contains more glucose than fructose. The

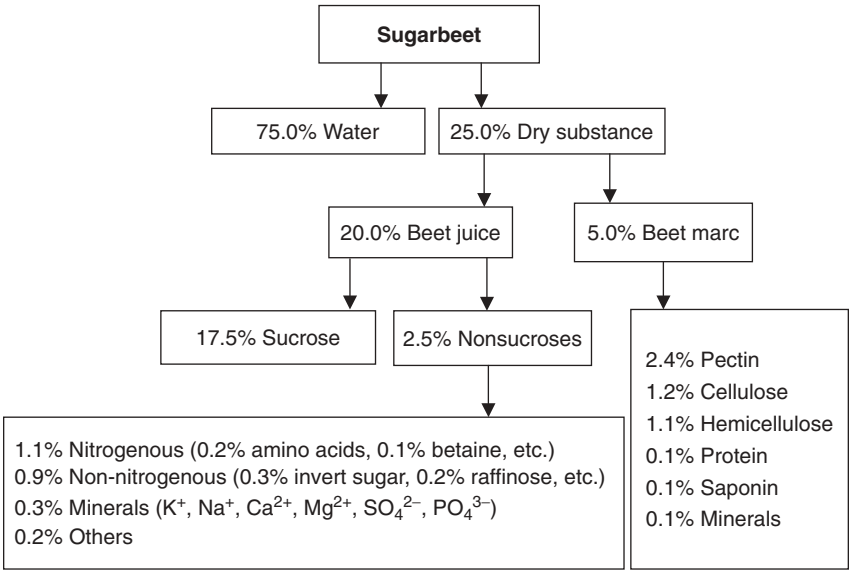


Figure 3.6 Composition of a typical sugarbeet

raffinose (a trisaccharide) content can vary largely depending on the location. Usually, sugarbeets with high sucrose content have less raffinose. The level of nitrogen in the fertilizer also affects the raffinose content (*the higher the nitrogen used, the higher is the raffinose content*).

Dextran and levan are the main polysaccharides present in sugarbeet juice. Their content increases when beets are damaged because microorganisms, particularly the *Leuconostoc group*, consume the sugar and convert some of it to dextran and levan. High contents of dextran and levan create difficulties during sugarbeet processing because of their colloidal nature (see Section 10 and 11 of this chapter).

Nitrogenous compounds (in amounts of 1 to 2%) are other components of beet juice. Almost all **amino acids** [compounds that contain an amino group (NH_2) and a carboxyl group (COOH), both groups joined to the same carbon] are present in beet juice and beet pulp (marc). Glutamine is present in the largest amount. Most amino acids are soluble in water and in alkaline solutions, so they do not precipitate in lime and end up in molasses. **Betaine** (in amounts of 0.1 to 0.2) is one of the nitrogenous compounds present in sugarbeet that end up in molasses. It makes up about 5% of the dry substance (*DS*) in molasses and is one of the most valuable components of sugarbeet. In the molasses desugaring by chromatographic (MDC) process, a considerable amount of high-purity betaine can be recovered (see Chapter 8).

Minerals in beet juice are referred to as ash. Analytical tests of sugarbeets have shown trace amounts of barium, boron, calcium, copper, lead, magnesium, molybdenum, nickel, selenium, silicon, and zinc.

Beet pulp (beet marc) consists of fibrous materials that are water insoluble and remain almost the same shape after the diffusion process. During sugarbeet growth in the field, some of the sugar is spent in producing pulp (the main component of cell walls) and the protoplasm.

During sugarbeet processing in the factory, the protoplasm is denaturized through the diffusion process (Station 7). The **denaturation process** changes the nature of the cellular protoplasm. Its main component (protein) is coagulated and made permeable. Heat and freezing are two **denaturants** (physical actions causing the denaturation process). In sugarbeet processing, the diffusion process uses heat. After the protoplasm is denatured, the cell is ready for the diffusion process. In this process, sugarbeet juice is separated from pulp and almost all the sugar and nonsugars stay in the diffusion juice. Therefore, the pulp remains almost sugarless.

The pulp content of sugarbeet ranges from 4 to 6%, but it can differ depending on the growing conditions and the variety of sugarbeet. Some scientists have proved that high-sugar-content beets contain higher pulp content as well (van der Poel 1998).

Pulp consists mainly of pectin, cellulose, and hemicellulose:

- **Pectin:** A gel-type substance that is insoluble in cold water but soluble gradually in boiled water, and stable during the diffusion process
- **Cellulose:** A wood-type substance that is insoluble in hot water, diluted acids, and alkalis solutions, and stable during the diffusion process
- **Hemicellulose:** A wood-type substance that is insoluble in hot water but soluble in hot diluted acid solutions, and stable during the diffusion process

Sugarbeet pectin has lower gelling power than apple or citrus pectin, because the molecular mass of sugarbeet pectin (15000 to 50000) is smaller than apple or citrus pectin (70000 to 90000).

Cellulose is a polysaccharide made of several thousand molecules of glucose ($C_6H_{12}O_{11}$). We humans cannot digest cellulose and hemicellulose because we do not have an enzyme that can break the polymer's chain. However, cattle, for example, have bacteria containing this enzyme in the stomach. The bacteria convert cellulose and hemicellulose into small digestible molecules that can be used by animals as a nutrient. (Pulp is a good food ingredient for some animals.)

Based on the beet's composition, the purity (P) of the **absolute beet-juice** (juice produced by squeezing beet-cossettes, or sliced beet) can be calculated from its sucrose content (S) and dry substance (DS) content:

$$P = \frac{S}{DS} \times 100 = \frac{17.5}{20.0} \times 100 = 87.5\%$$

A juice with 87.5% purity means that 100 kg (or any mass units) of dry substance of this juice (after all its water has evaporated) contains 87.5 kg of sugar and 12.5 kg of nonsugars.

Unfortunately, the diffusion process (Station 7) cannot selectively choose sugar and remove it from the beet cells. Instead, both sugar and nonsugars are subject to relocation, ending in the diffusion juice. If we could remove only sugar and leave the nonsugars where they belong, the purity of the diffusion juice would be much higher than it is now (usually 85 to 88%), which would make sugarbeet processing much faster and easier. As mentioned, the main goal of the sugar factory is to separate nonsugars from sugar to improve the beet-juice purity to the extent that sugar with 100% purity is produced. Such improvement is not achievable in one stage. In sugar technology, at each stage (called a station) a section of the factory is assigned to do a particular job.

Postharvested beets piled in beet storage contain some **tare** (clay, sand, stone, and trash mixed with the beets). The impurities that make up the tare differ depending on a field's properties, the harvesting method, and the size of the beets, all of which make it difficult to estimate beet-pile density. Following is the mass and density of a typical washed beet (beet without tare):

Mass	0.5–2.0 kg (1–4 lb)
Density	1050–1100 kg/m^3 (66–69 lb/ft^3)
Bulk density	650–700 kg/m^3 (41–44 lb/ft^3)

CHAPTER
3

SECTION 1
BEET RECEIVING AND STORAGE

SUBSECTIONS

- Beet Unloading and Sampling
- Beet Tare
- Beet Storage
- Sugar and Mass Losses during Storage
- Operating Problems

ABOUT THIS SECTION **This section** explains **Station 1** (Beet-Receiving and Storage) of sugarbeet processing. The beet-receiving and storing steps of Station 1 (see Figure 3.1.1) are the important starting points in sugarbeet processing. This station consists of unloading sugarbeets from the vehicle and piling them in the beet-storage area, where they remain until processing.

Beets delivered to the factory contain tare (impurities), which has to be separated from the beets during early steps of the process. **Beet tare** consists mostly of soil stuck to the beets after they are removed from the ground. Other components in beet tare are clay, sand, stone, and trash (leaves and weeds). Under good harvesting conditions, the tare varies from 2 to more than 8% on beet (OB). If growers harvest beets in wet weather, the amount of soil can increase significantly.

Sugarbeets are delivered to the factory mostly by truck or by railcar (in some areas). Farmers who are close to the factory might bring beets by tractor. In Europe and the United States, most beets are delivered by trucks because of the economical advantages over rail transport. The size of the load is an important factor in the cost of transporting beets (the cost decreases as the size of the load increases). (Transporting cost is about one-third of harvesting costs.) Because larger loads are more cost effective, larger trucks usually add a trailer. A large truck can carry about 25 t of beets, and a truck pulling a trailer can deliver up to 50 t of beets.

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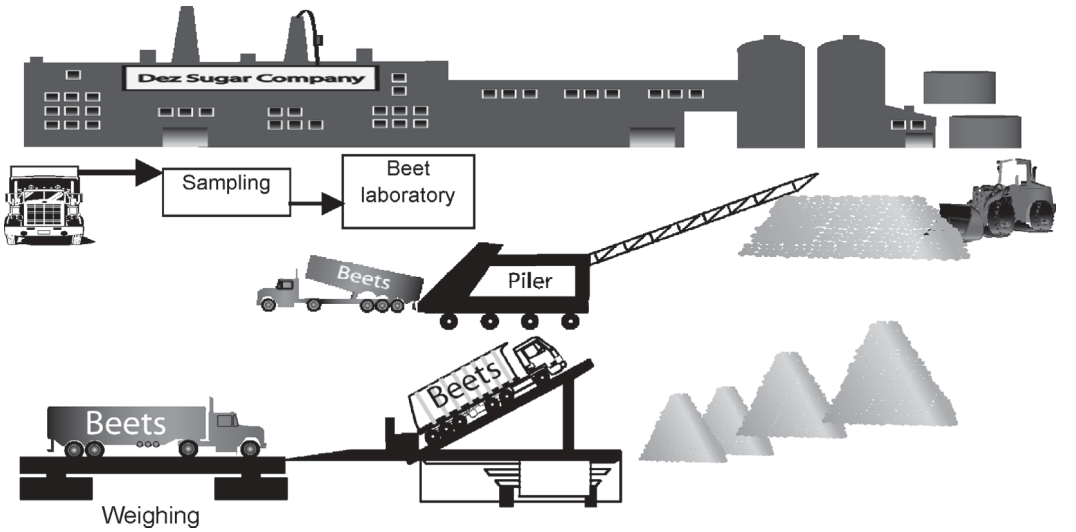


Figure 3.1.1 Flow diagram of beet receiving and storage (Station 1)



Figure 3.1.2 Beet receiving and piling at night

During harvesting, beets are delivered day and night (Figure 3.1.2). Delivered beets are then piled by pilers in the beet-storage area or discharged directly from trucks into the beet-receiving hoppers.

The beet-receiving and beet-storage operations are different in different countries because of differences in climate. Therefore, planning beet delivery to the receiving station, particularly in those areas with **short-period storage** (due to high temperatures) is of utmost importance.

BEET UNLOADING AND SAMPLING

Beets arriving at the factory are unloaded into the receiving hoppers or piled on the beet-storage area. Two types of unloading systems are available:

- Wet unloading
- Dry unloading

Wet unloading is not popular today because of high water usage and damage to beets if extensive high-pressure equipment is used. In wet unloading, beets are pushed from the vehicle into the flume by an overhead spray **nozzle** (firehose). The nozzles (Figure 3.1.3) are usually operated from the control room. The correct adjustment of water pressure is important in preventing beet damage, so a pressure of about 2.5 bar (36.7 psi) is usually used. The amount of water used is in the range of 225 to 550% on beet (van der Poel 1998).

Dry unloading is carried out in any of the following ways:

- Hydraulic side-dumping truck
- Hydraulic back-dumping truck
- Side-tilting platform
- Back-tilting platform
- Bottom-dump trailer

In the hydraulic side-dumping and back-dumping operation, hoisting equipment on the truck lifts the bed of the truck to unload the beets. In the side-tilting operation, the vehicle moves to a tilting platform. The platform is then raised on one side to unload the beets from the lower side of the vehicle. In the back-tilting operation (Figure 3.1.4), the platform is raised from the front and the beets are unloaded from the back of the vehicle. (Note: Bottom-dump trailers are not popular in beet transportation.)

Pilers are used to pile beets and are usually mobile. A beet piler consists of a swing-around receiving hopper that is positioned to receive the beets from the truck. It also contains a belt conveyor and a large boom (up to 30 m). Some pilers are further equipped with an automatic

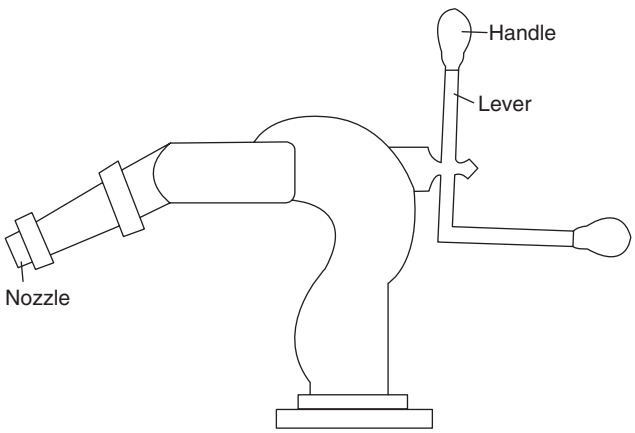


Figure 3.1.3 Spray nozzle

sampling device. A large piler can pile up to about 500 t beets per hour, so it can unload a 25 t truck in about 8 minutes.

As Figure 3.1.5 shows, a portable piler can handle the unloading operation from two trucks. After a truck is emptied, the receiving hopper swings to admit the next truck. From the hopper, beets drop onto the conveyor and are then lifted to another conveyor that carries them to a screen where soil and trash are removed. A larger conveyor (up to 30 m), called a **boom**, carries the beets to the storage piles. Sugar factories can pile beets 5 to 12 m (15 to 36 ft) high.

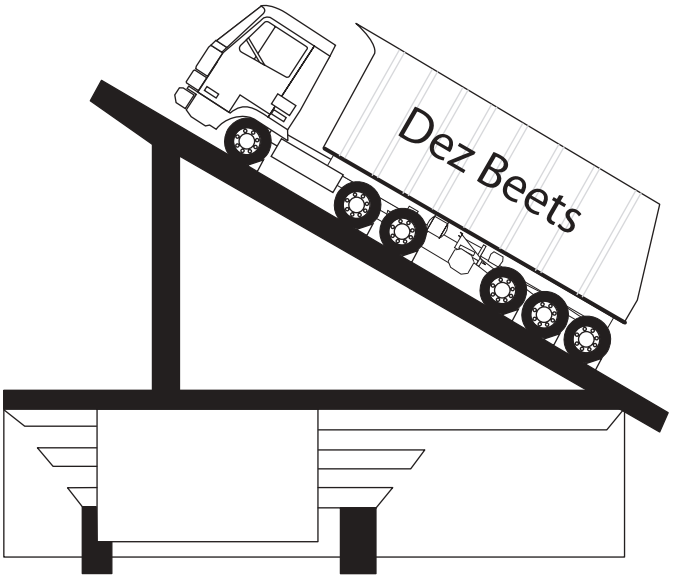


Figure 3.1.4 Back-tilting platform



Figure 3.1.5 Unloading beets from two hydraulic back-dump trucks by a piler

As the beets are passed over a screen, a portion of the soil is filtered out. The amount of removed soil is typically about 50% of the soil attached to the beets but particularly depends on the following:

- Soil content
- Soil moisture content
- Size of the piler's screen
- Speed of the piler's screen
- Nature of soil (claylike dirt is more difficult to separate from beets)

To determine the **beet gross weight** (weight of beets and beet tare) at the receiving station, each beet truck or railcar is weighed before unloading, and the weight (**weight-in**) is recorded by a computer. After unloading of beets, beet tare from the piler is transferred back to the truck and the vehicle is weighed again (**weight-out**). The beet gross weight equals the weight-in minus the weight-out (for detailed calculations, refer to the Beet Laboratory and Growers' Payment section of Chapter 2).

Beet sampling is performed at the receiving station by a sampling device that is installed after the weighing equipment. In some factories, sampling is taken before the delivery vehicle is weighed. Beet sampling is performed automatically or manually from sugarbeet loads. In the automatic method, a tube (called a sample tube) is lowered through the beet load to take a sample of about 10 to 12 kg (20 to 20 lb), which is sent to the beet lab. In general, the sampling method, sample weight, and frequency of sampling differ from factory to factory, based on the contract between the sugar company and the sugarbeet growers.

NOTES

- **In many countries, the growers' payment (see Chapter 2) is based on the beets net weight and the average sucrose content. Therefore, the accuracy of the weighing scales (weightometer or weigh-bridges) for weighing the beet-delivery vehicles is of utmost important. The scales are usually checked on a regular basis by a certified independent company. The scales usually differ based on the type of the beet-delivery vehicles (trucks or railcars).**
- **The method and accuracy of the analyses provided in the beet lab are also important, so extreme care is taken throughout the analyses to ensure the integrity of the tests.**

BEET TARE

Beets delivered to the factory contain **tare** (impurities) that has to be separated from the beets during early steps of the process. Beet tare consists mostly of soil stuck to the beets after they are removed from the ground. Other components in beet tare are clay, sand, stone, and trash (leaves and weeds). Under good harvesting conditions, the tare varies from 2 to more than 8% OB. In clay-type soil areas or if farmers harvest beets in wet weather, the tare can increase significantly.

The tare is measured in the **tare laboratory** (tare lab) by weighing the gross sample before washing (or brushing) to remove all nonbeet materials in the sample and the net after the tare removal. The results of the beet lab are used to determine the growers' payments for the delivered beets. The tare is usually calculated in percent on the **beet net weight** (*weight of beets free*

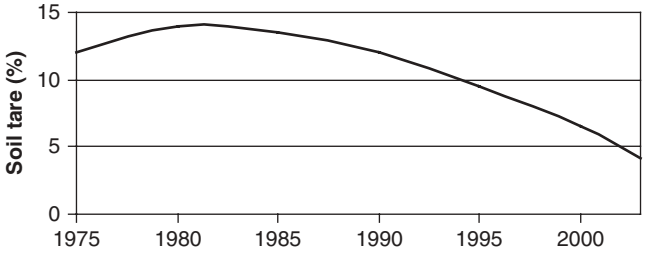


Figure 3.1.6 Decrease in soil tare in Swedish sugar factories

of dirt and trash with the crowns topped in a specified way) delivered to the factory. However, in some factories the beet **gross weight** (**weight of beets and beet tare**) is used for the calculations. The amount of tare depends on the following factors:

- Climate
- Soil type
- Soil moisture
- Type of harvester
- Harvesting method
- Loading techniques
- Type of storage floor (concrete or bare floor)

For the factory, high tare means increased processing costs in terms of water consumption, wastewater treatment, and damage to equipment. Therefore, factory managers pressure beet growers to reduce tare, particularly soil tare. Some sugar factories set a norm for tare. For example, the amount of soil, sand, and stone should not be more than 10% and trash should not be higher than 1% OB. Some factories require the farmer to share the costs of tare disposal if the tare exceeds the limit, and give a bonus for under-limit tare. Figure 3.1.6 shows the decrease in soil tare in Swedish sugar factories (IIRB 2005).

BEET STORAGE

Delivered beets are piled and stored in **beet-storage areas** (beet piling grounds). Four types of beet storage are available:

- **Factory beet storage:** This type of storage is located in the factory. is open and differs in size (a few thousands to hundreds of thousands of tons of beets). Monitor factory in Michigan has one of the largest storage areas, holding about 0.5 million t of sugarbeet (Figure 3.1.7). Factory beet storage is used for short- and long-period storing.
- **Remote beet storage:** This type of storage is located near the factory (between the fields and the factory). is open, and medium to large size. It is used for long-period storing until the beets are gradually transferred to the factory storage. Remote beet storage is popular in the United States and Russia.
- **Clamp beet storage:** This type of storage is located in the beet field, is open, and is of small size. It is used for short-period storing during the harvest, until the beets are gradually delivered to the factory. Clamp beet storage is popular in most parts of Europe.

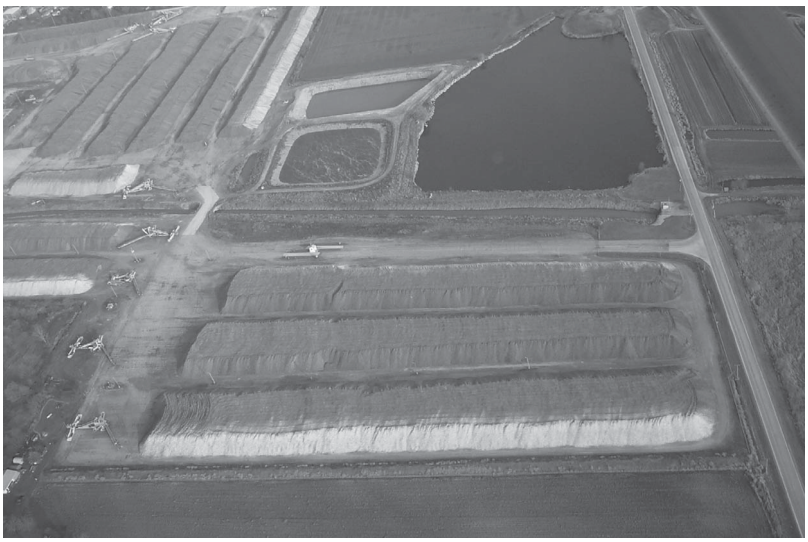


Figure 3.1.7 Monitor factory beet storage (Courtesy Michigan Sugar Company)

- **Deep-freezing storage:** This type of storage is located in the factory, is open or closed, and is of medium to large size. It is used for long-period storing. **Forced-air** ventilation is used to cool and deep-freeze the beet piles. Deep-freeze storage is popular in the United States, Russia, and in north part of Iran, where weather conditions permit.

In open storages, sugarbeets are kept in piles about 300 m (1000 ft) long and about 5 to 12 m (15 to 36 ft) high. Piles are placed with consideration give to wind direction and hours of natural sunlight. Some factories take aerial photographs of the beet piles every so often using special camera equipment that detects **active hot spots** (areas with higher temperatures).Active hot spots clearly show areas on beets that deteriorate more rapidly than others. Hot beets are removed from the piles for immediate processing.

For short-distance transportation of the beets in the storage area, usually large rubber-tired front-end loaders (with about 7 m^3 bucket capacity) and trucks are used.

SUGAR AND MASS LOSSES DURING STORAGE

Sugar loss during storage is any sugar-content reduction that occurs from the time the beets are weighed at the delivery to the storage and the time they are reweighed during processing (usually after beet slicing). Postharvested beets are still alive and continue to consume sugar. The losses result from

- Beet respiration
- Microorganisms

The sugar loss during beet storage occurs in a two-step process. First, part of the sucrose ($C_{12}H_{22}O_{11}$) is decomposed to produce invert sugar:

$$C_{12}H_{22}O_{11} + H_2O \rightarrow C_6H_{12}O_6 + C_6H_{12}O_6$$

Then the invert sugar is decomposed to produce carbon dioxide (CO_2), water, and energy:

$$C_6H_{12}O_6 + 6O_2 \rightarrow 6CO_2 + 6H_2O + \text{Energy (674 kcal)}$$

Later **during processing**, part of the invert sugar will decompose to some acid (e.g., lactic acid) and the coloring substances will negatively affect the process. Invert sugar formation during storage can be high (100 to 200 g per ton beet per day under normal storage conditions). The intensity of sugar loss during storage depends on the following factors:

- Microorganisms
- Temperature
- Dirt content

The intensity of sugar loss mainly depends on the temperature (*the higher the temperature, the higher is the sugar loss*). In general, stored beets need protection from high temperatures and frost during storage to reduce sugar losses. The worst that can happen is for beets to go through **freeze-and-thaw** conditions. The risk of freeze-and-thaw is highest during the late campaign when unusually warm weather can thaw the frozen beets and they are left under warm conditions for a long period before being processed.

The **freezing point** is related to the concentration of the solute (*the higher the solute concentration, the lower is the freezing point*). Because of a high dry\-substance concentration, sugarbeet freezes below 0° (at about –7°C). At this point, respiration stops because the beet cells are no longer active. The following preventive actions can be taken to reduce sugar losses in beet piles:

- **Using forced-air ventilation:** Is helpful during early and late stages of storage. During the early days of storage, when the temperature is high, the forced air removes the respiration heat from the piles, and in the later storage can deep-freeze the beets when the weather temperature reaches below 0°. In this system, several ducts (channels) are installed under the piles and air is forced through the piles by blowers.
- **Covering beet piles with plastic:** Is practiced in areas with very cold winters to protect the beets from freezing during storage.
- **Deep-freezing the beets:** Is used in open and closed storages. The closed storages are known as **beet sheds**, one of those is shown in Figure 3.1.8. Factories in North Dakota and Minnesota (both in the United States) use deep-freezing in closed buildings with concrete floors. A building size of 50 m by 250 m and 15 m high can hold about 70000 t of beets.

Because of sugar loss during storage, the factory must process the beets as soon as possible. In countries where the average daily temperature is above 30°C (86°F), such as countries in North Africa, southern Asia, and southern Europe, **long-period storage** is not possible and sugarbeets are processed within a few hours to a week of delivery to prevent high sugar loss. In these areas, harvesting is usually extended over a three- to five-month period, and growers deliver their beets according to a schedule. On the other hand, in northern Europe and North America harvesting time is much shorter (four to six weeks), and beets can be stored up to six months at low temperatures. In either case, substantial **sugar-loss during storage** is unavoidable.

Figure 3.1.9 (Vukov 1977) shows the effect of temperature on sugar losses during storage. The typical sugar loss is 100 to 200 g per ton beets per day (0.01 to 0.02 % OB), under normal



Figure 3.1.8 Loading frozen beets in a beet shed (Courtesy, Minn-Dak Farmers Cooperative Sugar Company)

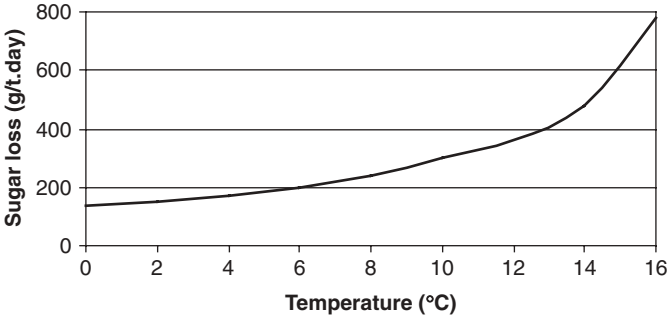


Figure 3.1.9 Effect of temperature on sugar loss

storage conditions and up to 2 kg on unfavorable conditions (note that numbers are just indicative). Figure 3.1.10 shows the sugar losses (% OB) caused by microorganisms from the L-lactic acid in the diffusion juice (van der Poel 1998).

Sugar loss per day per ton beet (m_S) related to temperature (T) can be estimated by the following equation (Vukov 1977):

$$m_S = 140 \times 10^{0.0343 \times T} \tag{3.1.1}$$

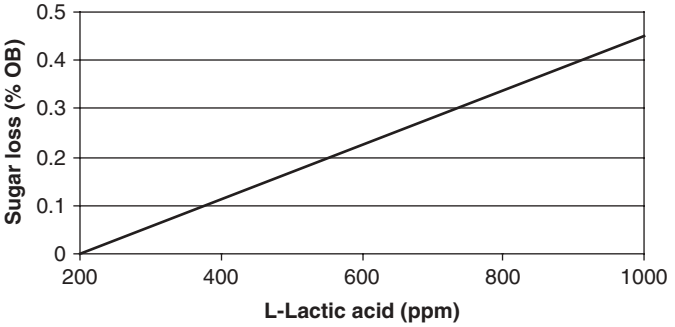


Figure 3.1.10 Sugar loss caused by microorganisms arising from lactic-acid production

EXAMPLE

Given:

Operation capacity of the factory	8000 t beets/day
Beet-sugar content	17.0%
Temperature at beet-storage area	5°C

Calculate:

Sugar loss per day per ton of beet
Sugar loss per day (% on beet)
Sugar loss per day per ton of sugar
Sugar loss per day (% on sugar)
Sugar loss (t/day)

$$M_S = 140 \times 10^{0.0343 \times 5} = 160.8\,\text{g/d per t beet}$$

$$160.8 \times \frac{100}{10^6 g/t} = 0.016\%\ \text{OB}$$

$$160.8 \times \frac{17.0}{100} = 27.3\,\text{g/d per t sugar}$$

$$27.3 \times \frac{100}{10^6} = 0.003\%\ \text{OS}$$

$$27.3 \times \frac{8000}{10^6\,\text{g/t}} = 0.22\,\text{t/d}$$

NOTE

In sugar terms, sugar (sucrose) conversion (inversion) to invert sugar during beet storage and beet processing is considered sugar lost. The formation of invert sugar not only decreases the efficiency of the factory but also increases the amounts of some nonsugars, which can contribute to operational difficulties. The invert sugar (as a nonsugar) later decomposes to acids and coloring substances that negatively affect the processing. For simplicity, scientists consider only lactic acid formation as a base for sugar-loss calculations.

Mass loss during storage occurs from the time the beets are weighed at the delivery to the storage and the time they are reweighed during process (usually after beet slicing). The mass loss during storage is caused by beet respiration and microorganisms.

Chemically, beets lose weight during storage mainly from dehydration (water loss) caused by respiration. This type of reaction is called a **dehydration reaction** (a reaction in which one or more molecules of water are released during respiration). During storage, the beet loses not only sugar and water but also pulp (marc), so its mass is reduced. The decrease in the pulp content of sugarbeet can be up to 10% (Vukove 1977). Here again, the intensity of mass loss depends mainly on temperature (*the higher the temperature, the higher is the mass loss*) and the length of storage. The amount of mass loss varies significantly (100 to 600 g per ton beet per day or 0.01 to 0.06% on beet per day) depending on temperature and humidity. Normally, the mass loss is more intense during the early days of storage.

Physically, beets also lose weight during **beet fluming** (the transport of beets by force of water) and washing, before reaching the slicing station. During fluming and washing, small **beet particles** from the damaged beets remain in the flume water. The amount of mass loss depends on the efficiency of the equipment employed in these stations. The amount of mass loss during fluming and washing is in the range of *0.2 to 0.6%* on beet (OB).

OPERATING PROBLEMS

The **problems** that can occur during beet-receiving and beet-storage operations depend mainly on storage conditions.

High Sugar Loss during Beet Storage

Under normal storage conditions, the typical sugar loss per day is 0.01 to 0.02% OB. High sugar loss during storage is usually caused by one or more of the following:

- **High temperature:** Beets lose more sugar at higher temperatures, so they should be kept at the lowest possible temperatures (*the optimum temperature is 1 to 3°C*).
- **Time:** Beets have a high sugar loss during the first 30 days of storage and late in the campaign when beet piles start deteriorating due to freezing and thawing.
- **Microorganisms:** Microbes (mainly mold and bacteria) attack damaged beets, which therefore have the highest sugar loss.
- **High dirt content:** Beets with more dirt lose more sucrose during storage, so it is best to clean beets before storing.
- **Damaged beets:** Damaged beets are attacked by microorganisms more than normal beets and suffer higher sugar loss.
- **Frozen-thawed beets:** Thawed beet tissue loses its ability to hold onto cellular juice (thawing tears cell walls), so the juice leaches out, greatly increasing the sugar loss.
- **Wrong topping:** Topping is the cutting done to the crown of the root. The amount of crown removed varies in different areas. The beets should be cut below the green leaf stalks of the epicotyl (see Chapter 2). In general, sugar losses during storage increase when beets are cut below or above the normal level.
- **Long-period storage:** Beets have high sugar loss toward the end of a long campaign when the weather starts to warm up. Sugar factories can experience unplanned longer campaigns if problems occur during beet processing, causing shutdowns.

- **Cutting beets at the wrong level**: The beets should be cut uniformly below the green leaf stalks of the crown. Cutting the beets at the right level leaves a cut surface area of about 5 cm (2 in.) in diameter. In general, sugar losses during storage increase when beets are cut below or above the normal level.
- **Wilted beets**: Beets that lack water become wilted (plant disease by fungous) during harvesting and storing. Wilted beets are susceptible to more damage and sugar loss during storage.

CHAPTER 3

SECTION 2

BEET DRYCLEANING

ABOUT THIS SECTION **This section** discusses **Station 2** (Beet Drycleaning) of sugarbeet processing. Station 2 (Figure 3.2.1) includes a hopper, conveyor, and a large vibrating screen. The equipment is installed outside the main processing building next to the flume system. (Older beet-sugar factories do not have a drycleaning station, but most newer factories in North America and some newer factories in Europe do.)

Before being processed, beets are cleaned to remove tare. **Tare** consists of stones, sand, soil, clay, trash, and beet crowns mixed with the beets. These impurities cannot be removed in one step; several processes are required to clean the beets before they enter the slicing station.

At the beet-drycleaning station, the beets are precleaned without using water just before being washed in the beet washer. The purpose of this station is to remove any beet tare that could otherwise enter the flume system and the beet washer with the beets so that less wash water is needed later in the beet-washing process. The drycleaning operation is a valuable environmental benefit and a cost savings for the factory.

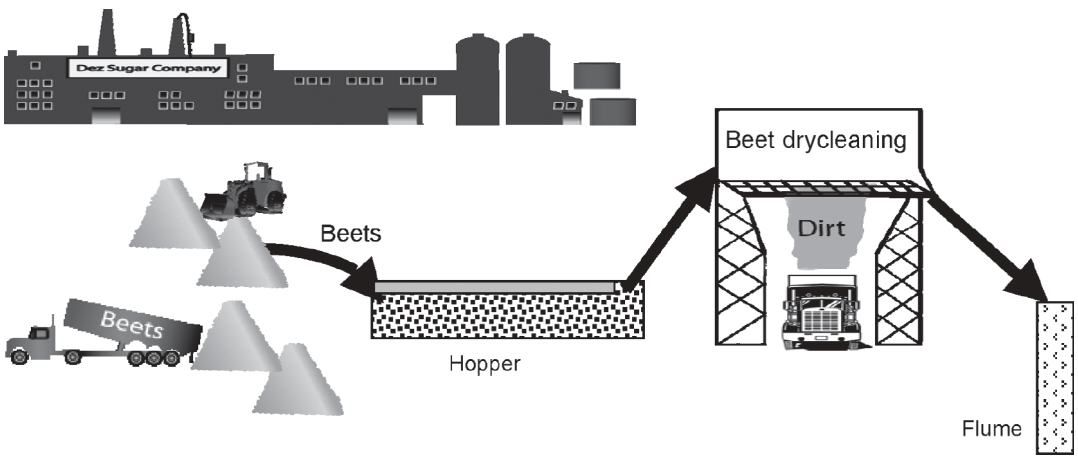


Figure 3.2.1 Flow diagram of beet drycleaning (Station 2)

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Figure 3.2.2 Beet-drycleaning equipment

The beet-drycleaning (dry-screening) **station** is used in some modern beet-sugar factories to separate stone, sand, and part of soil from the beets. Any **clay** (moistened soil) stuck to the beets cannot be separated by the drycleaning system. Materials larger than about 12 mm (0.5 in.), such as large stones and weeds, are also excluded from the separation at this station. But loose soil, sand, small stones, beet tops, and leaves can be separated from the beets by their screening. The separated materials are usually trucked to the field.

In the drycleaning station, the beets are first transferred from the storage area into the beet hopper using front-end loaders or trucks. Beets can also be unloaded into the hopper of the drycleaning belt directly from the truck (see Figure 3.2.3) or rail car when they are delivered to the factory. Note: In some European factories (e.g., a few in France), the drycleaning is used prior to beet storing. This is possible because the beets are delivered to the factory gradually due to the **short-period storage** (few days). (Note: In factories with short-period storage, planning for harvesting the beets and their delivery to the factory is based on a strict schedule.)

From the hopper, the beets are elevated by a conveyor to a vibrating screen. Here, part of impurities is separated from the beets by **vibration** (shaking). Then, a belt conveyor delivers the drycleaned beets to the flume channel.

In the drycleaning station, three types of screening systems are usually used:

- Cable screen
- Grab-roller screen
- Spiral-roller screen



Figure 3.2.3 Beet unloading from a hydraulic back-dump truck into the drycleaning hopper

All three types are based on the vibration process and are self-cleaned by a contact mechanism between the rubber rollers. The grab-roller type has slots of 11 to 16 mm (0.4 to 0.6 in.) between the rubber rollers. Slots are kept clean by a special mechanism.

OPERATING PROBLEMS

The **beet-drycleaning station** provides cleaner beets to the beet washer, so less wash water is needed in the beet-washing process. This is an environmental advantage and a cost savings for the factory. However, a negative aspect of the beet-drycleaning system is the partial separation of **beet chips** (small beet particles), which becomes part of sugar loss. The beet chips left after beet precleaning can be removed from flume water by a vibrating chip-separator, which is explained in Section 5 of this chapter.

Technologically, the operation of the drycleaning station is not complicated and is almost problem free. The biggest issues are probably screen maintenance for wear and breakage of the screen cables to maintain good chip separation performance. The screen issue can be solved by routine inspections and maintenance as needed. This is a relatively simple task, requiring only a short downtime to replace cables as they wear out. If a grab-roller screen is used, any in place repair of the roller or a screen change requires a longer downtime.

CHAPTER
3

SECTION 3

BEET CONVEYING AND FLUMING

SUBSECTIONS

- Beet Conveying
- Beet Fluming
- Operating Problems

ABOUT THIS SECTION **This section** covers **Station 3** (Beet Conveying and Fluming) of sugarbeet processing. The beet conveying and fluming system of Station 3 (see Figure 3.3.1) uses dry or wet systems to transport beets from the storage area to the next station (stone and trash separation). The most popular method of conveying is **beet fluming**.

In this section, both the dry and wet conveying (transport) systems and beet flume are described. You will also become familiar with their operating problems.

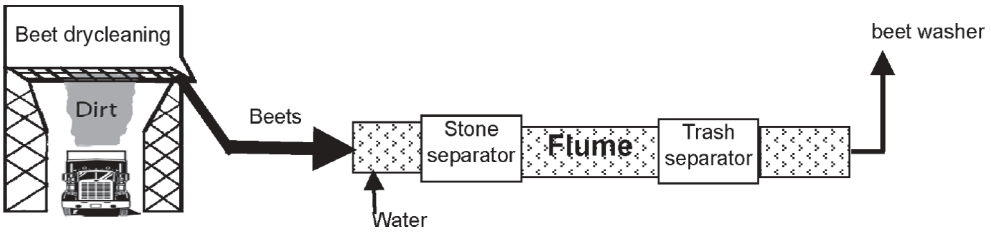


Figure 3.3.1 Flow diagram of beet conveying and fluming (Station 3)

In the factory, beets are transferred from **beet storage** (beet-piling ground) or the drycleaning station (if the factory is equipped with this station) to the beet flume. The **beet flume** is a U-shaped channel for transporting sugarbeets by the force of water.

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Figure 3.3.2 Beet fluming

BEET CONVEYING

Two types of beet-conveying systems are available:

- Dry conveying
- Wet conveying

Dry Conveying

In a **dry-conveying system**, front-end loaders transport the beets from the factory beet storage to the beet hopper which is installed in front of the beet flume.

The dry system offers the following advantages over wet transportation:

- Less loss of sugar during transportation
- Less water usage

However, dry conveying damages beets more than fluming.

Wet Conveying

In a **wet-conveying system**, a truck or a loader transfers the beets from storage into beet bins to be moved to the main flume system (see Figure 3.3.3). The beets delivered directly to the

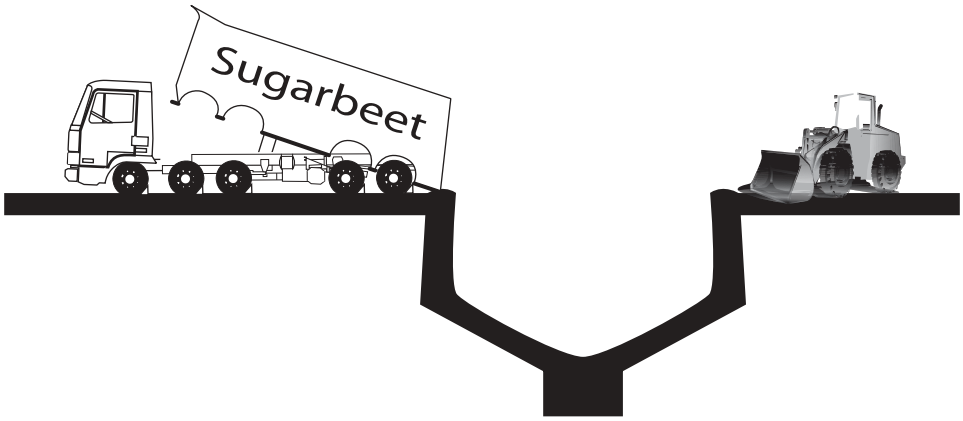


Figure 3.3.3 An underground beet bin

factory are unloaded from the truck or rail car into the bins. The beet bins are built underground or above ground. The bin walls are built at a 45° angle to permit the beets to drop into the bin channel, at the bottom of the bin. The bin channel is about 0.5 m (1.5 ft) wide and 0.5 m deep. Water is pumped into the channel to move beets to the main flume in which the stone and trash catchers are installed. Beet fluming is a help to the beet washer because some of the dirt is washed away during fluming, so the beets are partially cleaned before reaching the beet washer.

BEET FLUMING

The **beet-flume system** (water-transport system) transports the beets to the stone and trash separators by the force of water. Fluming is possible because the sugarbeet has a density slightly greater than that of water (beet density is about 1.1 t/m^3).

Fluming requires water in the range of 300 to 500% on beet (OB). To save water and to control the quantity of mud, flume water is treated for mud separation in the factory's wastewater ponds, and the treated water is returned to the flume. Some factories use a **flume-water clarifier**, which allows several returns (recycles) of the water to the flume, while part of the water is discharged to the ponds at each cycle. In the clarifier, the majority of the mud and solids are removed. The flume-water clarification reduces considerably the amount of discharged wastewater of the factory. (More explanations about flume-water clarification are provided in Section 5 of Chapter 3.)

The beet flume (see Figure 3.3.4) is made of concrete or sheet metal; its U-shaped bottom has a slope of about 1.5%. Flumes are 1.0 to 1.3 m (3 to 4 ft) wide and about 1.0 m deep, depending on the processing capacity of the factory. Modern flumes are built with a bypass that allows the fluming to continue if a plug occurs.

The fresh water used in fluming is generally **condenser water**. During fluming, high-pressure water should not be used because it damages the beets.

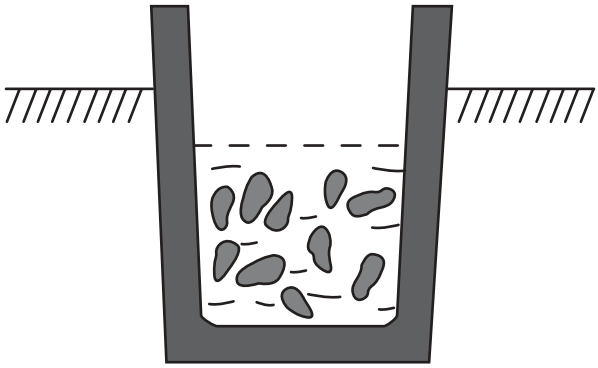


Figure 3.3.4 Beet flume

OPERATING PROBLEMS

The **problems** that can occur during the beet conveying and fluming include high sugar loss and slow fluming.

High Sugar Loss during Fluming

It is unlikely that healthy beets lose much sugar content by contact with water in the flume or beet washer because sugar removal from the beet cells requires a **denaturation process** (destruction of cell walls). The denaturation process requires high temperatures (about 70°C) and long retention time, and these conditions do not exist during the beet-fluming and beet-washing process.

Under normal operating conditions (healthy beets and a flume water temperature of around 20°C), the loss of sugar to flume water should not exceed 0.4% OB. The causes of high sugar loss are the following:

- **Damaged beets:** Damaged beets are softer, so they are broken more easily than undamaged beets, causing higher sugar loss.
- **Frozen beets:** More sugar leaches out of the cells of frozen beets compared with normal beets because freezing tears cell walls.
- **Water temperature:** For fresh beets, the water temperature in the flume should be kept between 20 and 30°C. For frozen beets, the temperature should be as low as possible so that it will not cause the beets to thaw. In general, thawing damaged beets in the flume and in the beet washer increases sugar loss.

Slow Fluming

The normal retention time of beets in the flume to reach the washing station is one to four minutes, depending on the flume length. For fluming, a flow speed of about 0.8 m/s is needed. A slow speed can cause sand to settle at the bottom of the flume. The usual causes for slow fluming are the following:

- **Wrong ratio of water to beet:** Fluming requires water in the range of 300 to 500% on beets present in the flume. Excessive quantities of beets can block the flume in sharply curved areas, which causes the water to spill over the sides of the flume. Beets should not be damaged while fluming if the ratio between the water and the beets is held in a reasonable range.
- **Inconsistent beet processing:** When slowing down beet processing, the ratio of water to beet should be kept in a constant required range (300 to 500%). In other words, the flume operation should be adjusted by the rate of beet processing. Slower fluming causes sand to settle in the flume.
- **Low water pressure:** The pressure of the fluming water should not be less than 15 psi (1 atm). High-pressure water should be avoided because it damages the beets.
- **Wrong flume dimension:** The flume might be undersized. A beet flow of 300 t/h needs a flume width of 0.8 m (2.6 ft). In addition, flumes should not have sharp curves.
- **Incorrect incline:** The flume should have a slope of about 2 to 3%, and in curved portions, the slope should be a bit greater.
- **Low speed:** To prevent sand settling and to ensure beet movement, the speed of the flume flow should not be less than 0.8 m/s (2.6 ft/s).
- **Dirty beets:** Factories that receive beets with high soil or mud content may face a slow flume flow because of mud settling in the flume. In these situations, the flume should be cleaned more often. Some factories use high-pressure overhead nozzles to flush mud from the bottom of the flume. Others use cable hoist plows to manually pull the mud along the flume to where it can be flushed.
- **Frozen beets:** Frozen beets may cause problems in some places in the flume. Some factories try to increase the speed of the fluming by increasing the temperature of flume water (by mixing in warm water from different parts of the factory). This practice speeds up the flow in the flume but increases sugar loss due to higher water temperature.

CHAPTER 3

SECTION 4

STONE AND TRASH SEPARATION

SUBSECTIONS

- Stone Separator
- Trash Separator
- Operating Problems

ABOUT THIS SECTION **This section** explains Station 4 (Stone and Trash Separation) of sugarbeet processing. Station 4 (see Figure 3.4.1) includes the stone and trash separation equipment which are installed outside the main processing building next to the flume system. The mixture of beets and water in the flume goes through the stone and trash separators for removing stones and trash. These coarse impurities must be removed completely to prevent damage to the beet slicers and other processing equipment of the factory.

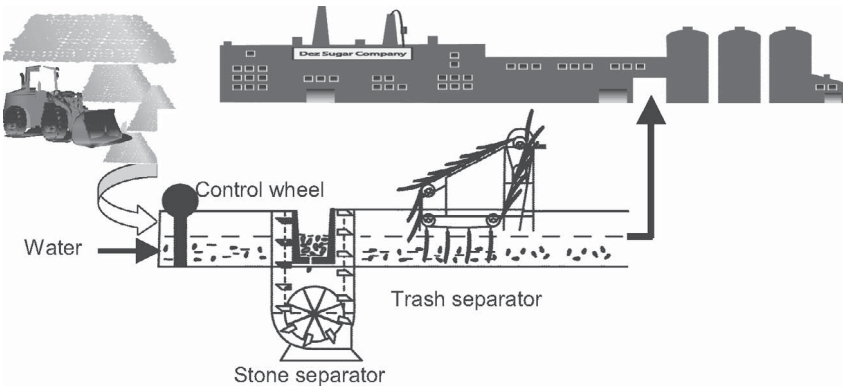


Figure 3.4.1 Flow diagram of stone and trash separation (Station 4)

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The separation of coarse impurities from the beets consists of two steps:

- Stone separation
- Trash separation

STONE SEPARATOR

Stone separators (stone or rock catchers) are installed around the flume system to separate large stones (rocks) from beets. The stone separator operates based on the **density difference** between beets and stones. In a water–beet–stone mixture, stones have higher density than water and beet, causing the stones to drop and separate from the beets in the stone separator. Beet-sugar factories use three types of stone separators:

- Bucket stone-separator
- Drum stone-separator (Dyer type)
- Belt stone-separator

The **bucket stone separator** (see Figure 3.4.2) is made up of a vertical pipe connected to the bottom of the flume with several **openings** (pockets). A pump applies an upward flow of water through the openings to prevent beets from falling through the spaces. Therefore, when the beets reach the openings, they remain in suspension and stones fall into the openings. The separated stones are then removed by the bucket conveyor, and water runs into the flume channel. The pressure of the pump should be adjusted so that it is a little greater than the pressure of the flowing water in the flume. For example, if the water in the flume flows at a speed of 0.8 m/s (2.6 ft/s), the speed of the upward flow should be adjusted to about 0.85 m/s (2.8 ft/s).

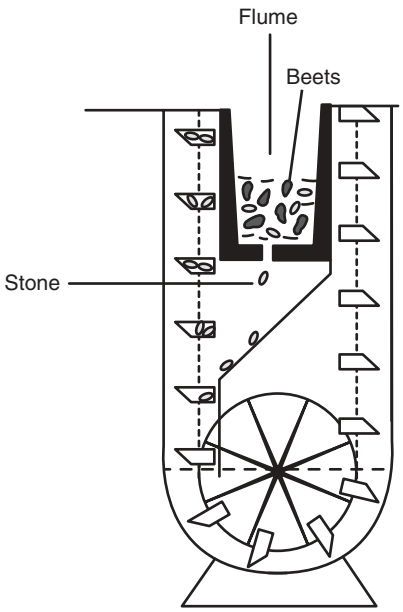


Figure 3.4.2 Bucket stone separator

The **drum stone-separator** is welded to the sides of the flume channel. It consists of a drum with pockets on the sides. The continuous rotation of the drum causes the beets to pass over the pockets while the stones fall into the pockets for removal. Modern drum separators are capable of removing up to 99% of stone, sand, and gravel without the loss of any beets.

The **belt stone-separator** consists of a collecting device for separating stones and a belt for removing the separated stones. A pump adjusts the speed of the water to keep beet in a floating (buoyancy) condition.

NOTE

New factories usually use two or three stone separators (sometimes one after the trash separator) to ensure complete stone removal.

TRASH SEPARATOR

Trash (sugarbeet leaves and weeds) can clog the slicer's knives, plug the diffuser, and bring many impurities into the process. Therefore, the flume system contains a trash separator to remove trash flowing with the beets. A number of types of trash separators are used in sugar factories.

In the **rake trash separator** (see Figure 3.4.3), the trash is caught on rakes that move against the flow of the flume in an endless chain. The speed of the chain is a slow speed of about 0.2 m/s. The trash is released from the rakes as the rakes are tilted outside the flume.

In general, trash separators can remove a large part of trash but not small trash and **beet chips** (small beet particles). Beet chips and small trash can be removed by using a **vibrating chip separator** (explained in the next section), which is usually installed in modern factories after the trash separator or the beet washer.

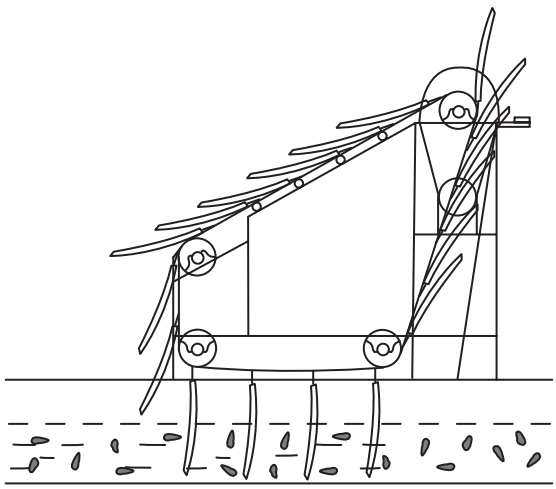


Figure 3.4.3 Rake trash separator

OPERATING PROBLEMS

The **complete separation** of trash and stones from beets during beet fluming is important to the operations of the later stations. Sometimes, the separation equipment is unable to handle unusually large quantities of stone, trash, and rotten beets. In that case, the beets on the beet conveyor running into the hopper of the slicing station are inspected manually by a worker. The place where the worker is positioned to pick the remaining impurities is called the **picking table**, a characteristic of old beet-sugar factories. (The human factor is always essential for perfect sugarbeet operation.)

CHAPTER 3

SECTION 5

BEET WASHING AND FLUME-WATER TREATMENT

SUBSECTIONS

- Beet Lifting to Beet Washer
- Beet Washer
- Flume-Water Treatment
- Operating Problems

ABOUT THIS SECTION **This section** is about **Station** 5 (Beet-Washing and Flume-Water Treatment) of sugarbeet processing. Station 5 (Figure 3.5.1) includes beet-lifting, beet-washing, and wash-water treatment equipment.

In this section, you learn about the types of beet washer, the beet-chip separator, and the flume-water treatment system.

Beets that have been separated from stones and trash and partially washed during fluming are now moved to the main processing building of the factory by fluming. Once inside the factory building, the beets are lifted from the fluming channel to feed the **beet washer** for the **final cleaning**. (Note: In some factories, the beet washer is installed in open area next to the trash separator, and the washed beets are transferred to the main building by a conveyor to feed the beet slicers.) In the beet washer, soil and clay stuck to the beets and sand are washed away before the beets enter the slicing process (Station 6). The beet washer not only cleans the beets but also removes most of the microbes coming with the beets.

The main job of the **beet-washing station** is to supply the slicing station with a steady flow of clean beets. The beet washer is the last point for removing remaining tare, so its operation is important to the performance of the further stations. In many ways, the beet-washing station controls the slice rate of the factory because any stones or excess weeds can damage

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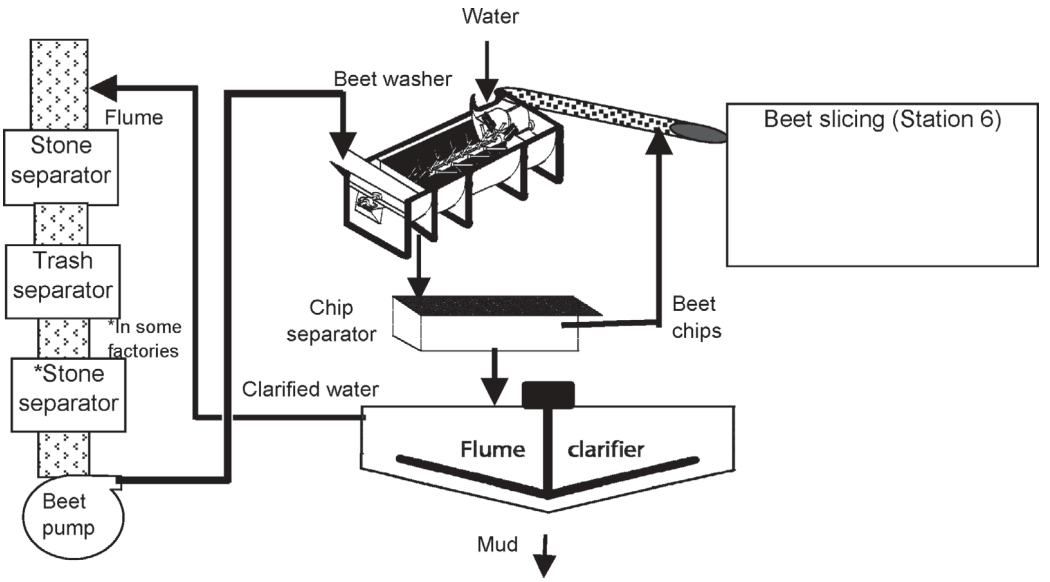


Figure 3.5.1 Flow diagram of beet washing and wash-water treatment (Station 5)

slicer knives and cause delays in beet slicing. Therefore, the washing station must be maintained in a way that ensures that enough (but not overfilled) clean beets are always in the slicer's hopper. In modern factories, beets are cleaned in two separate washers.

Water discharged from the beet washer and from the flume system contains:

- Small beet particles (chips)
- Small trash particles
- Mud

In older factories, the wash water from the beet washer and flume system is pumped to a **mud-settling pond**. The upper flow from the pond is mixed with makeup water (usually condenser water) to be reused in the fluming and beet washer.

In modern factories, the wash water is first sent to the **chip separator** to separate beet chips and trash particles from the water. The separator removes the heavier beet chips from trash particles. After separation, the beet chips are sent to the beet hopper and then to the **slicers**, and trash particles are sent to the **pulp presses** to be pressed with pulp. The water from the chip separator is discharged to the wastewater treatment system to be cleaned and reused in the flume and beet washer.

BEET LIFTING TO BEET WASHER

Once inside the factory building, the beets are lifted from the fluming channel into the beet washer. The beet washer is located several meters higher than the flume to make the delivery of washed beet to the slicers easier. Beets are lifted to the beet washer by one of the following means:

- Lifting wheels (rotating elevators)
- Bucket conveyer
- Belt conveyor
- Pump

The most common way of feeding the beet washer is by way of the beet pump for three good reasons:

- It has lower initial investment
- It is easy to maintain
- It needs less room

However, the beet pump is energy intensive and can damage the beets slightly, thus raising the sugar loss. **Beet conveyors** have been used recently by some factories to deliver sugarbeets to the beet washer. The belt is made of a one-piece reinforced rubber, 600 to 1200 mm (24 to 48 in.) wide with capacity up to 500 t/hour. The belt conveyors can handle up to a 30° incline and both horizontal and vertical curves. Some suppliers offer conveyors with multipoint drives with up to 240 m (800 ft) length. (Note: The conveyors generally have lower installation and maintenance costs than beet pumps.)

The amount of wash water needed for fluming and cleaning beets is high (about 200 to 500% OB), depending on the beet-handling and cleaning system of the factory. Fortunately, most of this water is recycled to the flume system after soil and organic substances (mostly sugar lost in the washing and fluming process) are removed in the factory's wastewater ponds. The water that has to be discharged is equal to 10 to 20% OB (depending on the wastewater system of the factory).

BEET WASHER

The three types of beet washers commonly used in sugar factories are the following:

- Arm washer
- Drum washer
- Spray washer

Arm Beet Washer

The **arm beet washer** (see Figure 3.5.2) is an old type of washer, consisting of a cylindrical drum with either a single or a double shaft with multiple arms. The bottom of the tank is perforated. The shaft rotates at about 5 RPM (revolution per minute). Beets enter from one side, are gradually washed by rubbing against one another as they are moved by the arms, and are discharged from the other side. The water enters the washer and moves against the beets; therefore, the water that contacts the clean beets is fresh. Immediately after leaving the washer, the beets are usually sprayed with very clean water. Arm washers cause mechanical damage to beets, so they increase the sugar losses. They also need a lot of maintenance.

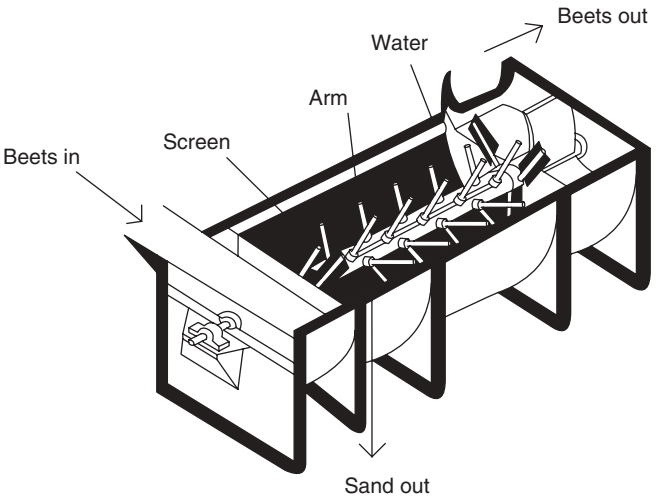


Figure 3.5.2 Arm beet washer

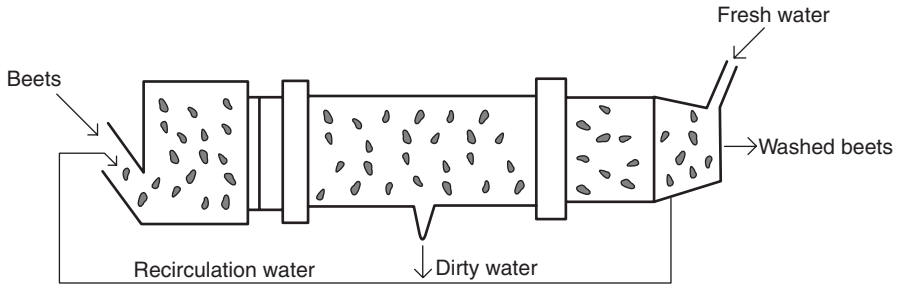


Figure 3.5.3 Drum beet washer

Drum Beet Washer

The **drum washer** (see Figure 3.5.3) is the most advanced type of beet washer. The drum rotates on a few track rollers. The rollers are mounted on a steel frame. A drum washer 3 m in diameter and 20 m long can wash about 8000 t beets per day. The drum is usually filled to 40% of its capacity and rotates at 3 to 5 RPM. A newer drum washer that is 3 m in diameter and 10 m in length with two shafts can wash up to 20000 tons of beets per day. Figure 3.5.4 displays a Maguin medium-size drum beet washer.

Spray Beet Washer

The **spray washer** is often used in factories where beets are cleaner upon arrival, usually in areas with a dry climate. This washer is also used as a finishing beet washer in factories where beets are not very clean. The washer consists of several rotating rolls (72 or more) across the length of a bar and a sprayer that constantly sprays high-pressure water to clean the beets. The beets enter at the high end and move toward the low end by the rotating rolls and by gravity.

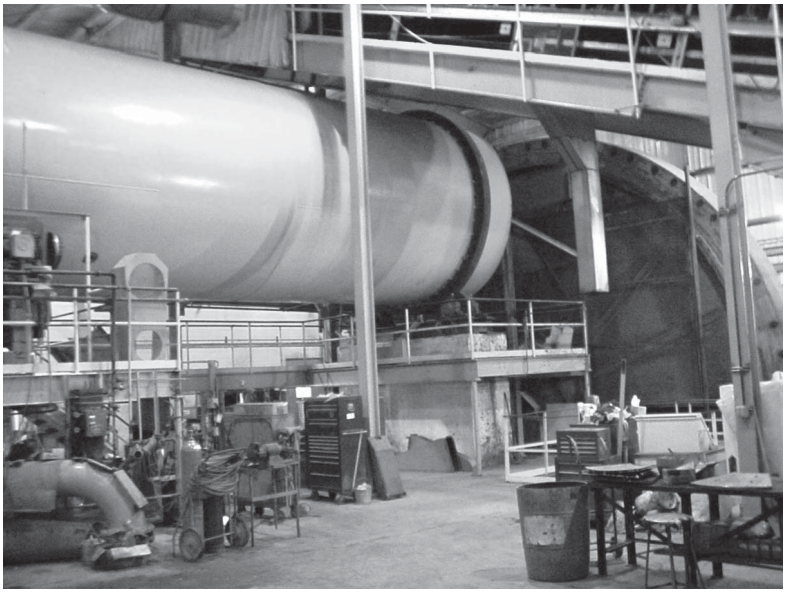


Figure 3.5.4 Maguin beet washer (Courtesy, Minn-Dak Coop.)

The rolls vibrate the beets to release loose dirt and rub the beets against each other to release dirt from the beets.

FLUME-WATER TREATMENT

A coarse metal **grate** (a screen with large openings) is installed at the end of the beet washer to separate water from the beets. The beets are then sprayed with fresh water. Depending on the factory design, the flume water discharged from the beet washer is cleaned in different ways. Some factories have a chip-separation system. In these factories, the flume water is directed to the **chip-separation system** and then to the wastewater-treatment system. In factories without the chip separator, the flume water is directly sent to the wastewater system.

Chip Separation from Flume Water

Beet chips are small, broken particles of beets that could not be separated in the earlier stages of operation. Modern beet-sugar factories are equipped with a **chip separator** that removes beet chips and small trash particles (weeds) left in the water discharged from the beet washer. The separator is a **drum screen filter** consisting of a vibrating screen (see Figure 3.5.5) with openings of about 0.2 mm, followed by a **separating belt** that separates the heavier beet chips from weeds by a rolling mechanism (chips have the ability of rolling, while weeds do not). After separation, the beet chips are sent by a conveyor to the beet hopper, which feeds the **slicers**. Weeds are sent to the **pulp presses**. Chip separation has the following advantages:

- It increases sugar recovery by recovering beet chips
- It decreases the load of solids entering the factory's wastewater system
- It increases the factory's income by recovering beet chips and trash that would otherwise be wasted

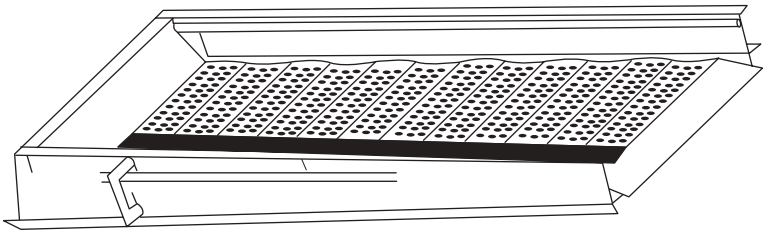


Figure 3.5.5 Screen of chip separator

Mud Separation from Flume Water

Used wash water from the beet washer or from the chip separator contains **mud** (fine and watery soil) as well as dissolved sugar leached from beets during fluming and washing. The flume water is sent to the **mud-separation system** (mud-settling pond, flume clarifier or mud centrifuge), where the mud, sand, and trash (weeds) residue are separated.

In older factories, only a **mud-settling pond** takes care of the mud in the flume water. Muddy water from the beet washer and flume system is pumped to the mud-settling pond, which has a short retention time (several days). The upper flow from the pond is mixed with fresh water (usually condenser water) to be used in the beet flume and beet washer. In these factories, when the mud pond becomes full of mud, another pond is used.

In modern factories, the mud-separation system consists of the following elements:

- Flume clarifier
- Mud-settling pond
- Flume filter (trash filter)
- Cake filter (usually filter press or centrifuge filter)

In this system (see Figure 3.5.6), muddy water first goes to the **flume clarifier**. The clarifier uses the density difference between dirt and water for separation. The overflow from the clarifier is clean enough to be sent to the fluming and washing system. The underflow from the clarifier, which contains mud and fine trash materials, is sent to the mud-settling pond and from there to the **flume filter** (trash filter) to remove any trash and fiber material from the mud and to prevent it from entering the watertreatment system. The mud is then filtered by a **filter press** or **centrifugal filter** to produce cake to ease its handling process. (Note: Some modern factories are not equipped with all of the elements listed above, particularly the last one).

The material recovered by the trash filter is returned to the pulp presses to become part of the pulp production from the factory. Water from the trash screen is sent to the flume pond and then to the wastewater treatment system. Trash separation prevents the mud ponds from producing odor (see Chapter 10).

The **flume clarifier** (see Figure 3.5.7) is used to remove mud from flume water. It consists of a large tank with a shallow cone bottom that is 50 to 100 m (150 to 300 ft) in diameter and 2 to 4 m (6 to 12 ft) deep. The clarifier contains a slow-moving rake driven from a central shaft. There are arms connected to the rake. The feed enters the center of the clarifier, and the heavier particles flow downward and gradually make a mud layer where the clear liquid stays on top. The clear water spills over the edge of the tank into a trough. The rake arms gently move the mud to the center of the tank, where it flows through a large opening and is pumped by the mud pump.

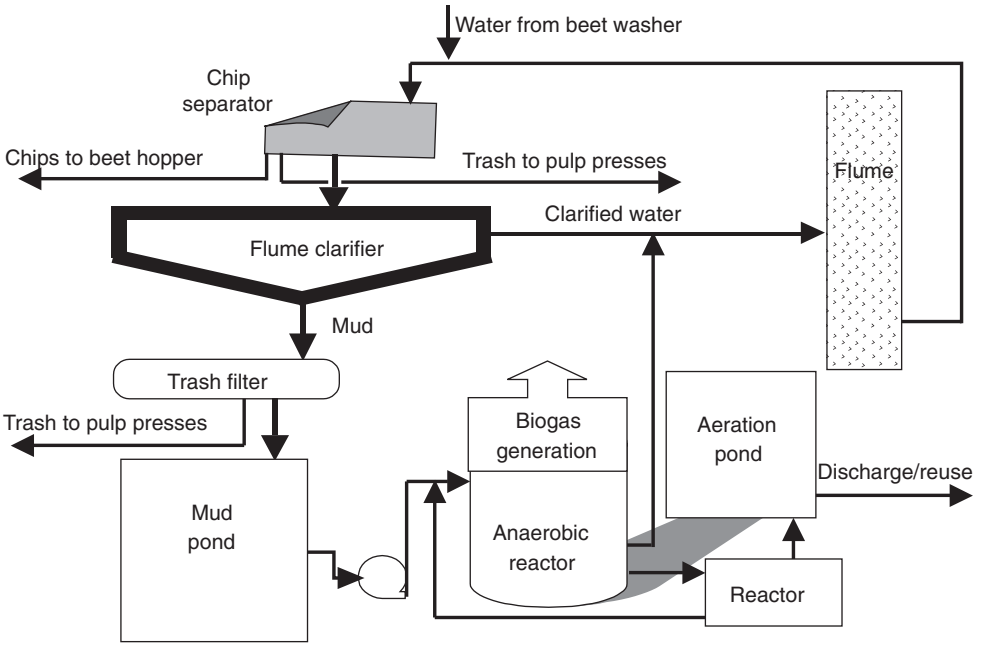


Figure 3.5.6 Flow diagram of a typical flume and wash-water treatment

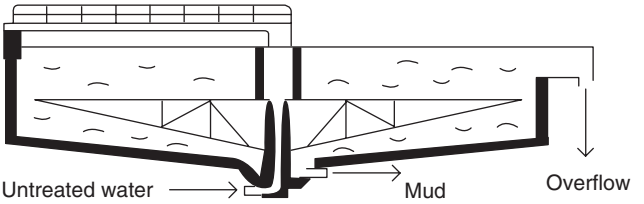


Figure 3.5.7 Flume clarifier

Water Consumption for Beet Fluming and Washing

Between 200 and 500 t of water is required to flume and wash 100 t of beet. Most of this water is supplied by various in-factory sources such as condensate, condenser water, and wastewater treatment system. Fluming and washing water are almost free of organic compounds and contain mostly suspended solids. Therefore, these waters are treated in the wastewater system of the factory and are recycled for fluming and washing beets.

OPERATING PROBLEMS

The **problems** that can occur during the beet washing are insufficient beet cleaning and damage to the beets.

Insufficient Beet Cleaning

The usual causes of insufficient beet cleaning are the following:

- **Insufficient wash water:** A typical drum beet-washer needs about 150% wash water on the mass of sugarbeet (% OB) to clean them. (*Washed beets should not contain more than 0.2% soil.*) Excessive amounts of trash and weeds can plug and blunt the slicer knives, causing difficulties and producing poor quality cossettes. This results in low sugar recovery during the diffusion process and problems during purification.
- **Insufficient beets in the washer:** An incompletely filled washer cannot properly wash the beets because the beets do not rub against each other. The required fill level depends on the washer and is usually provided by the supplier.
- **Abnormal (misshaped) beets:** Clay can stay in the pocket of beets that have an irregular shape. These beets are more difficult to wash.
- **Dirty wash water**
- **Extremely dirty beets**
- **Undersized beet washer**

Damaged Beets

The possible causes of damaged beets during washing are as follows:

- **High elevation between the beet pump and the washer:** The elevation between the beet pump and the washer should not exceed 20 m (60 ft); otherwise, beets may break and get damaged.
- **Use of improper type of beet washer**

Problems with Chip Separation from Flume Water

The chip separation is a situation of constant optimization. As the dirt and trash load varies, adjustment of the gravity chip-separation system is required to maintain optimum dirt removal and chip recovery. When the dirt is muddy, there is often a buildup of dirt at the separation point. This can cause more chip to be diverted to trash and ultimately the separation system to be shut down for cleaning.

NOTES

- **Stations 1 through 5 explained the process of sugarbeet receiving, piling, storage, and cleaning. The equipment belonging to these stations is installed outside the main building of the factory, with one exception being the beet washer is inside in older factories. Therefore, the operations of these stations is called yard operation. From the next station (Beet Slicing), the main operation of the sugarbeet processing starts. After leaving the beet washer, the beets are directed to the hopper of the beet slicer to wait for the slicing process.**
- **Spraying beets with fresh water after discharge from the washer is important to remove microorganisms from the surface of the beets. Even so, some microbes remain in the pockets and crevices of beets. When beets are sliced in the next station (Beet Slicing), the microbes are distributed through the cossettes and will continue into the diffuser (Station 7) causing sugar loss (see Section 7 for microbial activities in the diffuser).**

CHAPTER 3

SECTION 6

BEET SLICING

SUBSECTIONS

- Cossette Quality
- Beet Slicers
- Slicer Knives
- Knife Maintenance
- Cossette Conveyor
- Cossette Weighing Scale
- Operating Problems

ABOUT THIS SECTION **This section** discusses **Station 6** (Beet Slicing) of sugarbeet processing. Station 6 (see Figure 3.6.1) consists of the beet conveyor, beet hopper, slicers, and the knife-maintenance shop (for sharpening the slicers' knives). The equipment included in this station is installed inside the main processing building. In the slicers, beets are cut up into cossettes (see Figure 3.6.2).

The beet-slicing station is one of the most important stations in sugarbeet processing. Quality slicing depends on many factors, as are explained in this section. The operating problems that can occur in slicing operations will be also discussed.

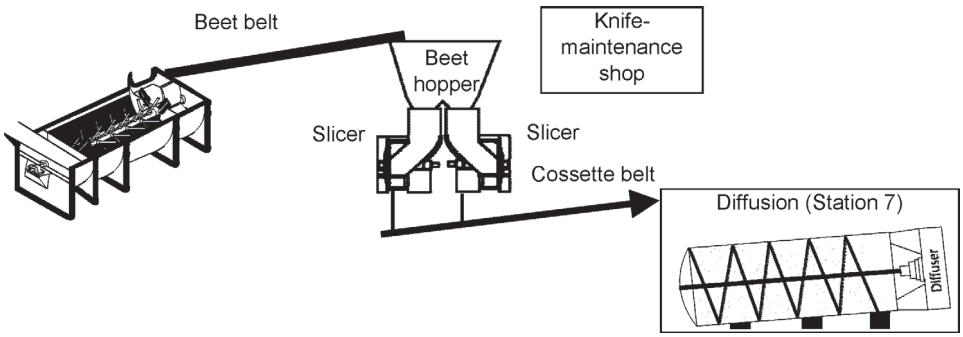


Figure 3.6.1 Flow diagram of beet slicing (Station 6)

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Figure 3.6.2 Beet cossettes

Slicing beets is the process of cutting beets into long, thin strips, called **cossettes**. In the slicing station, a conveyor (usually the belt type) continuously delivers clean beets from the beet washer to the **beet hopper** (a cone-shaped container) that feeds the beets to the slicers (see Figure 3.6.3). In the factories equipped with the **chip separator** (see Section 5 of Chapter 3), the separated beet chips are also sent to the beet hopper.

The main function of the beet-slicing operation is to improve the diffusion operation and the removal of sucrose from the beets. This is achieved mainly because of the increase of the surface area of the beets. Increasing the surface area maximizes the following:

- The contact area between the beet cells and the water in the diffuser (Station 7)
- The movement of sugar from the cells to the diffusion juice

The slicing process (Figure 3.6.4) is affected by the firmness of the beet tissue and the degree of **lignification** (the woody character of the cell walls of the beet). This characteristic of the beet is known as **slicing resistance** or **beet firmness**. The firmness depends on the beet's variety, pulp (marc) content, growing conditions, and particularly, storage conditions. For example, the firmness of frozen beets is two to three times higher than beets stored under normal conditions.

The beets must be sliced with sharp knives to minimize tearing (rupturing) of the beet cells. Torn cells allow more impurities (nonsugars) to be diffused into the surrounding juice, causing an increase in nonsugars and, consequently, a reduction in juice **purity** (sucrose content



Figure 3.6.3 Beet belt conveyor

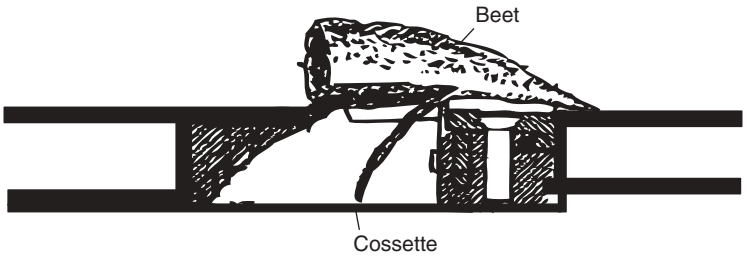


Figure 3.6.4 Slicing beets to cossettes

as % of dry substance). Tearing also creates more foam (because of saponin release) in the juice during diffusion, which is undesirable.

COSSETTE QUALITY

For complete removal of sugar by the diffusion process, the size and quality of the cossettes are important. Generally, for the same result, thick cossettes need more diffusion time, or a higher amount of diffusion water than thinner cossettes. Therefore, thin cossettes are desirable. However, there is a limit for cossette thickness. The desired characteristics for high-quality cossettes are as follows:

- Uniform width (3 to 6 mm thick, square or V shape)
- Uniform length (30 to 60 mm long)

- Minimal amount of fines
- Minimal amount of slabs
- Non-mushy texture

(Note: The term **slab** is used by sugar technologists to indicate several cossettes that are not separated from one another. The term **mushy** refers to cossettes that are softer and smaller than usual.)

In the factory, cossette quality is usually evaluated by three methods:

- Silin number (*SN*)
- Swedish number (*SWN*)
- Mush content (*MC*)

The **Silin number** (*SN*), named after its developer, P. M. Silin, determines cossettes quality. It is the length (in meters) of 100 g of cossettes. In this test, 100 g of cossettes are laid out lengthwise and the total length is measured. Cossettes shorter than 10 mm (0.4 in.), cossettes thinner than 5 mm (0.2 in.), and all slabs are separated and weighed separately. (The separated portion is called **rejects**.) Good cossettes have an *SN* of 10 to 18 m (30 to 54 ft). The mass of rejects should not be more than 5 g (not over 5%).

The **Swedish number** (*SWN*) determines cossette permeability. It is the ratio of the mass of cossettes longer than 50 mm to those shorter than 10 mm in 100 g of cossettes. Good cossettes have an *SWN* greater than 10.

EXAMPLE 1

Given:

Mass of cossettes longer than 50 mm	60 g
Mass of cossettes shorter than 10 mm	4 g

Calculate:
SWN

$$SWN = \frac{60}{4} = 15\,\text{g}$$

The **mush content** (*MC*) is the ratio of the mass of cossettes less than 10 mm long to the total cossette mass (100 g). Good operations require that the *MC* be smaller than 5%.

EXAMPLE 2

Given:

Mass of cossettes shorter than 10 mm	4.8 g

Calculate:
MC

$$MC = \frac{4.8}{100} = 4.8\%$$

BEET SLICERS

Slicers contain knives in **knife blocks** (knife boxes) for slicing beets to cossettes. The knife blocks are used to hold several knives in the drum of the slicer. Sugar factories usually use one of the following types of slicers:

- Drum slicer
- Disc slicer
- Centrifugal slicer

Drum Slicer

Modern factories use modified **drum slicers**. The new model of Putsch drum slicer has a horizontal shaft and a vertical rotating disc with a slicing capacity of 8000 t per day. This slicer has 22 knife boxes, each holding 6 knives. This machine is controlled by a dc or frequency-controlled AC motor.

Maguin (see Figure 3.6.5) is another type of drum slicer with a capacity of 3000 to 10000 t per day. Its new model with a double-knife block can slice 10000 t per day. The knife blocks are inserted into disc openings. Each knife box consists of one or two rows of three knives. The cutting length of the knife box is 600 mm. The beets enter the slicer at the top of the drum and contact the rotating knives at the bottom of the drum.

Both Putsch and Maguin drum slicers have an automatic knife-changing feature so that a block can be replaced with a new one with minimum stop time. The size of the cossettes can be changed by adjusting the guide plate.

Disc Slicer

Disc slicers have a vertical shaft and a horizontal rotating disc. The disadvantages of disk slicers are the following:

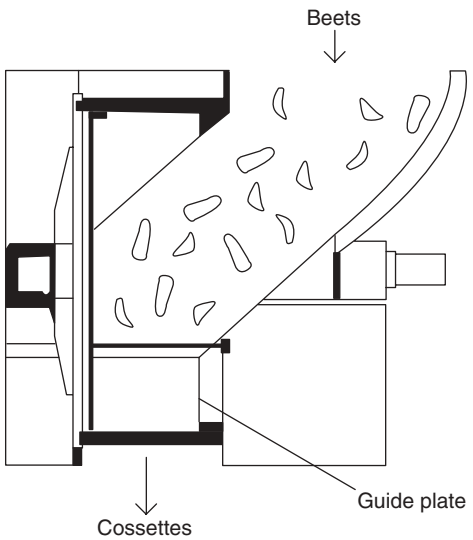


Figure 3.6.5 Maguin beet slicer

- The cutting speed between the outer and inner knife blocks is not equal across the radius of the disc (the inner knife blocks move faster), and this reduces the uniformity of the cossettes
- The quality of cuts depends on the pressure applied on the slicing disc by the level of beets in the beet hopper

Centrifugal Slicer

Centrifugal slicers are mainly used in some Eastern European countries. This type of slicer has a stationary vertical drum and rotating paddles inside the drum. Centrifugal force and the paddles press the moving beets against the stationary knives. The advantage of this type of slicer is that it is possible to change a knife block while the slicer is running. The disadvantage is that this slicer requires 20 to 30% more energy than the other types to cut a same amount of beets.

SLICER KNIVES

The most important factors in producing quality cossettes are the type of **knives** used and their position in the knife box. Most knives have a V shape. Each knife consists of several V-grooves, called a **division**. For example, a standard size knife 200 mm long has 36 divisions, so each division is 5.6 mm wide. The thickness of the cossette depends on the size of each division. The shape of the cossette depends on the position of the knives in the knife block.

Two types of knives are used, A and B (see Figure 3.6.6). The A-knife is finished at both ends with a half-V shape. The B-knife ends with a full-V shape. The most popular knives in beet sugar factories are Konigsfelder knives. These knives come with different numbers of divisions.

KNIFE MAINTENANCE

Well-maintained, sharp **knives** are important for producing quality cossettes. During operation, dull and damaged knives are separated and maintained in the knife maintenance shop as follows:

- Dirty knives are washed in hot water
- Bent grooves are straightened
- Damaged edges are ground (faced)

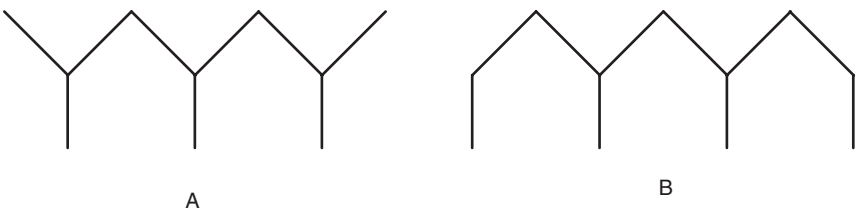


Figure 3.6.6 Slicer knives

- Edges are reground (jointed)
- Edges are sharpened with a file

COSSETTE CONVEYOR

A **belt conveyor** sends the cossettes to the diffuser (see Figure 3.6.7). The conveyor moves on two pulleys: a drive pulley and a take-up pulley. Belt conveyors (see also Section 3 of this chapter) need low energy and little maintenance. However, they operate with a low incline angle (up to 20°) and should not have a speed of more than 100 m/min. A conveyor belt 1.0 m (3 ft) wide can transport about 200 tons of cossettes per hour.

COSSETTE WEIGHING SCALE

The **cossette weighing scale** is one of the most important pieces of equipment in the factory because its correct functioning ensures a reliable material balance (see the Note below). Therefore, it should be checked often and adjusted when necessary. Sugar plants usually use a belt-conveyor scale, such as the Merrick scale, for weighing cossettes.

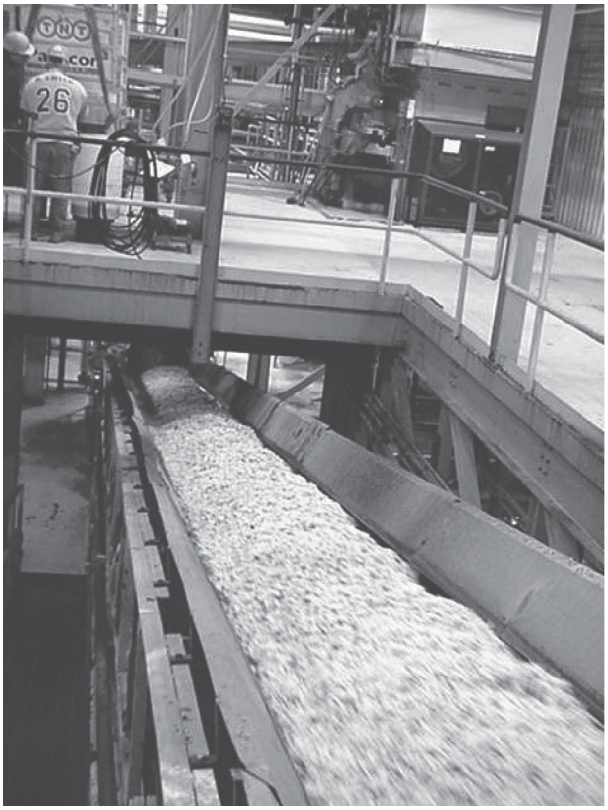


Figure 3.6.7 Cossette belt conveyor

NOTES

- **The amount of sugar in the beets entering the factory is calculated by measuring the total mass of cossettes processed in 24 hours multiplied by their average sugar content. Samples of cossettes are taken from the cossette belt at regular intervals (usually every two hours) to be tested for sugar content.**
- **The beet slicers must be frequently cleaned with steam to remove accumulation of cossette particles and microbes. Feeding the diffuser with cossettes containing as few as possible microbes is important in diffusion operations (the subject of the next section).**
- **It is important to observe cleanliness of the beets when they arrive the slicing station. The beets should be free of impurities (trash, sands, stone, pieces of metals, and rotten beets). Complete separation of impurities from beets during previous stations is important to operation of the slicing stations. As mentioned in Station 4, sometimes the separation equipment (stone and trash separators) and beet washer are unable to handle unusually large quantities of impurities. During these times, the beets on the beet conveyor running into the hopper are manually inspected for remaining impurities by a worker. The place that the worker is positioned to closely inspect the beets is called the picking table.**

OPERATING PROBLEMS

The **main problems** that can occur in the slicing operations are poor-quality cossettes and low slicer efficiency, which are covered next.

Poor-Quality Cossettes

Poor-quality cossettes are usually caused by one of the following problems:

- **Poor beet-washing operation:** Beets insufficiently washed reduce the quality of cossettes and increase knife maintenance.
- **Fresh beets:** Fresh beets and beets harvested during rainy days are brittle and break easily, creating a difficult job for the slicers. The factory faces this problem particularly during the beginning of the campaign.
- **Damaged beets:** Damaged beets or beets with high pulp (marc) cause the knives to **dull** frequently. In such cases, the knife clearance should be enlarged, or steam should be frequently injected into the knife boxes as the slicer is running. Cossettes produced from damaged beets have a high mush content. Damaged beets plug up the diffuser and lower the diffusion-juice purity, resulting in a high sugar loss to the pulp.
- **Low level of beets in the beet hopper:** Uniform distribution of the beet feed over the cutting knives requires sufficient pressure. In disc-type slicers, this pressure is proportional to the level of beets in the beet hopper. The pressure in drum and centrifugal slicers is not as dependent on the level of beets in the beet hopper as it is in disc-type slicers. In factories with disc-type slicers, the level of beets in the beet hopper should be kept at no less than 2 m (6.6 ft) from the bottom of the hopper to create enough pressure. Otherwise, the beets move over one another while they are on the cutting disc, resulting in mushy cossettes.
- **Dull knives:** The quality of cossettes depends on correct maintenance (straightening, grounding, jointing, and filing) of the knives.
- **Dirty knives:** Beets with high marc (beet fiber or pulp) content cause rapid plugging of the knives. In this situation, the knives should be cleaned with pressurized steam.

- **Dirty beets:** Stones may destroy the knives, leading to excessive knife changes and possible delays in the factory's operation.
- **Wrong positioning of the knife blocks:** To produce quality cossettes, the knives in the knife block must be set correctly.
- **Wrong disc speed:** The slicing capacity of the slicer is proportional to the speed of the disc or drum. If the slicing speed is too slow, the knives will tear the beet tissue and cut coarse cossettes.

Low Slicer Efficiency

Low slicer efficiency is caused by the following:

- **Slow speed:** Slow speed of the disc or drum is the main cause of low slicing efficiency. The slicing capacity of the slicer is proportional to the speed of the disc or drum. The optimum speed depends on the slicer's manufacturer.
- Poor-quality beets
- Wrong knife position
- Insufficiently cleaned beets

CHAPTER 3

SECTION 7

JUICE DIFFUSION

SUBSECTIONS

- Denaturation
- Factors Influencing the Diffusion Process
- Diffusion Water
- Adding Press Water to the Diffuser
- Adding Pressing Aids to the Diffuser
- Adding Antifoaming Agent to the Diffuser
- Adding Biocide to the Diffuser
- Types of Diffusers
- Product and By-product of the Diffusion Process
- Theory of Diffusion
- Diffusion of Damaged Beets
- Material Balance of the Diffusion Process
- Operating Problems

ABOUT THIS SECTION **This section** discusses the operations of **Station 7** (Juice Diffusion). Station 7 (see Figure 3.7.1) consists mainly of the cossette conveyor, diffuser, diffusion-water tank, and fittings and accessories. In most sugar factories, the diffusers are installed inside the main building.

Brewing tea in a teapot is a simple example of the diffusion process (**infusion** process, in the tea drinkers' language). Here is how:

- Place 2 teaspoons (tsp) of tea leaves in the infuser, which sits in the neck of a teapot.
- Pour in 2 cups of boiling water. It is essential that the water have plenty of oxygen to release the flavor, so use water that has just come to a boil.
- Keep the teapot covered for about 4 minutes to brew the tea.

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During brewing, the heat from the hot water destructs (denatures) the wall of the cells, causing tea soluble-components (e.g, flavors, antioxidants, colorants, and caffeine) to move (infuse) from the tea leaf's cells to the water at a rate of concentration of the tea components, and this gradually decreases over time. The cause (driving force) of the movement of components is the **concentration difference** between two phases: inside and outside the cells. The movement continues until the concentration of components in the tea leaf's cells and the surrounding liquid is equalized. Within a short time, the tea leaf will no longer contain any soluble components, just cellulose material.

A process similar to tea brewing occurs in the factory's **diffuser**, in which **sliced beets** (cossettes) are kept in contact with hot water for about an hour to diffuse the juice from the beet cells. The hot water (about 70°C) used in the diffusers destructs the beet cells to make the movement of diffusing components possible. As the water moves ahead, it collects sugar (sucrose) and nonsugars (nonsucroses) from the cossettes and becomes a concentrated impure sucrose solution, known as **diffusion juice** (raw juice). The diffusion juice (with 85 to 88% purity) is sent to the purification station (Station 10) for the removal of certain nonsugars (impurities). In the meantime, the cossettes in the diffuser gradually lose almost all (about 98%) of their sucrose and turn into **pulp** (desugared cossette). The wet pulp is sent to the next station (Pulp Treatment) to be pressed, dried, and pelletized and sold as a by-product of the factory.

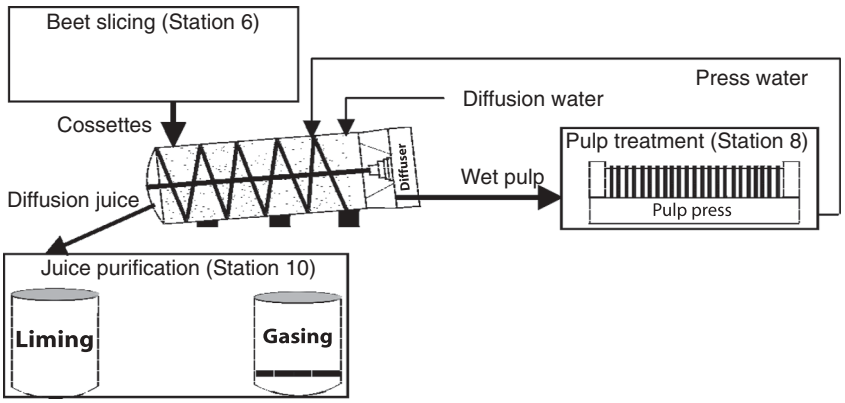


Figure 3.7.1 Flow diagram of the juice diffusion (Station 7)

In beet-sugar technology, the **diffusion process** is defined as the movement of diffusing components (sucrose and nonsucrose substances) from a higher concentrated side (inside the beet cells) to a lower concentrated side (outside the beet cells) through the cell protoplasm, which acts as a semipermeable membrane. The concentration difference between both sides of the membrane is the **driving force** (cause) of diffusion.

At the beginning of diffusion, the rate at which water molecules move through the cell wall from the water side into the juice side is high because there are much more water molecules in the water side than in the juice side. The result of the water molecules' movement is an increase in the volume of the juice side (now the juice inside the cell is diluted), which causes a buildup in pressure, known as **osmotic pressure**. At the same time, the heat in the diffuser

denatures (deforms) the protoplasm, causing it to become permeable, so components of the juice diffuse through the membrane because of the pressure buildup. The tendency of molecules to move decreases as the concentration difference between both sides of the cell wall decreases. Eventually, a time will be reached during which almost no molecules movement occurs. At that time, the system reaches **equilibrium**.

As a result of diffusion, almost all sugar (about 98%) and nonsugars are removed from sliced beets (cossettes) and accumulated in the water that was added to the diffuser. Diffusion improves under the influence of physical (e.g., temperature) and chemical conditions (e.g., pH).

The product of the diffusion process is **diffusion juice** and **pulp** is its byproduct. The pulp (desugared sliced beets) discharged from the diffuser is then pressed, dried, and stored.

Diffusion juice, made from average quality beets and correct diffusion operations, usually has about 15% dry substance and 0.5 to 1.5% insoluble substances. Its **purity** (sucrose content as % of dry substance) is 85 to 88%. Diffusion juice has a low purity (high nonsugar content) because, in the diffusion process (unlike the extraction process), the components are not chosen selectively (*both sucrose and nonsucrose substances are subject to relocation*). If just sucrose could be relocated, the purity of the diffusion juice would be much higher, and this would have a broad effect on sugar technology. Actually, some nonsucrose substances, such as KCl and NaCl, are relocated more easily than sucrose because of their lower molecular mass and higher **diffusion**



Figure 3.7.2 Tower diffuser installed outside the main building (Courtesy Minn-Dak Cooperative Sugar Company)

coefficient (as explained later). For example, 60 to 90% of K, Na, and Mg, 10 to 30% of Ca, and almost all amino acids and betaine are transferred to the juice.

A higher **diffusion efficiency** (degree of desugaring of the cossettes during the diffusion process, also known as **nonsugar elimination**; see the Appendix) is the main objective of the diffusion station. A higher efficiency results in a high-purity juice and low sugar loss to pulp. The **nonsugar elimination** (*NSE*) is the comparison (percentage) of the purity of diffusion juice with that of **absolute beet-juice** (juice produced by squeezing beet cossettes). (Note: The purity of the diffusion juice is higher than that of absolute beet juice because, during the diffusion process, most of the proteins and some of the minerals remain inside the cell and, consequently, exit the diffuser with the pulp.) Because some nonsugars are in the pulp, the diffusion-juice purity is increased as compared to absolute beet juice. The *NSE* of the diffusion process can be calculated by the following equation (see also the Appendix):

$$NSE = \frac{(P_2 - P_1)}{P_2(100 - P_1)} \times 10000 \tag{3.7.1}$$

EXAMPLE 1

Given:

Purity of absolute beet juice (P_1)	87.0
Purity of diffusion juice (P_2)	88.0

Calculate:

Nonsugar elimination in the diffuser (diffusion efficiency)

$$NSE = \frac{(88.0 - 87.0)}{88.0(100 - 87.0)} \times 10000 = 8.7\%$$

Usually, with undamaged beets, the *NSE* in the diffuser ranges from 2 to 10%. If the beets are damaged, the *NSE* can be as low as 0.

Sucrose in the beet cells is surrounded by a **cell wall** (protoplasm), and it does not transfer itself from the cells unless the three following conditions are met:

- Maximum contact surface
- Optimum temperature
- Denaturation

The first condition is covered by slicing the beets into small strips, called cossettes. This was the subject of the preceding section (Station 6). The necessity of the first and second conditions (surface and temperature) can be illustrated by an example. If an unsliced beet is soaked in cold water for a few hours, it does not lose its sucrose for a number of reasons:

- Only cells on the surface of the beet are in contact with the water
- The temperature is too low for the diffusion process to occur
- The cell walls are not destructed (denatured)

When a cossette, which has higher surface area than a whole beet, is in contact with cold water, the cell's protoplasm allows some molecules to pass. The cossette loses part of its sucrose

(incomplete diffusion) in cold water because, during the slicing, the walls of some cells are destructed. In addition, there is a larger contact area between the cossette and the water compared with an unsliced beet. Having said that, all three conditions should be met to achieve complete diffusion. The third condition, denaturation, is met by the **destruction** (deformation of the structure of a substance) of the cell's walls under chemical or physical conditions, which is called **denaturation** (changing the *nature*).

DENATURATION

Denaturation (changing the *nature*), in chemistry, means destruction (alteration) of the cell protoplasm by **coagulation** (see the Glossary) of its main component, protein, to make it permeable.

A **beet cell** consists of a cell wall, the protoplasm, and a vacuole (see Figure 3.7.3). The beet juice is in the vacuole surrounded by the protoplasm, which has a thin-membrane-type structure (2 micro meters thick). The protoplasm is **semipermeable** (a material that allows the passage of small particles but not large ones) to water and **impermeable** (nonpermeable) to sucrose and nonsucroses. Therefore, it prevents the diffusion process, unless a deformation in its structure occurs. After the protoplasm is denatured, it is permeable and ready for the diffusion process. Denaturation is accomplished by one of the following **denaturants**:

- Heat
- Freezing
- Chemical means

In beet processing, heat is generally used in the diffuser. It was found experimentally that it takes about 8 minutes at 70°C to denature 90% of the protoplasm of beet cells (Silin 1958). Figure 3.7.4 shows the relation between denaturation and time at different temperatures (Schneider and Hoffmann 1952).

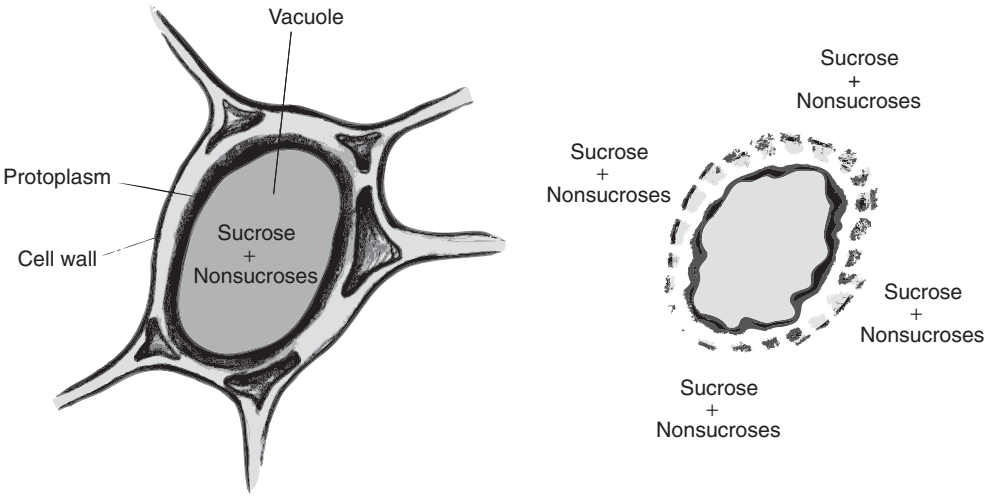


Figure 3.7.3 Cell wall shape before (*left*) and after (*right*) denaturation

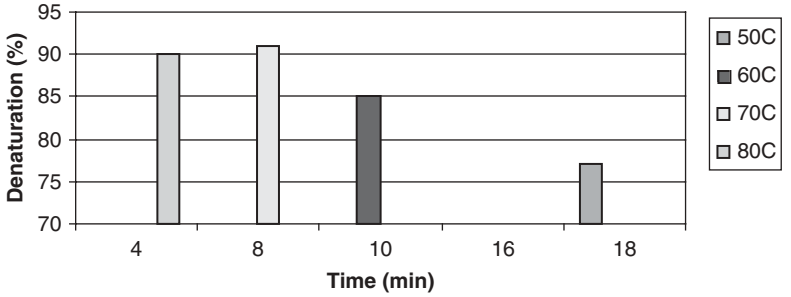


Figure 3.7.4 Denaturation of beet cells at different temperatures

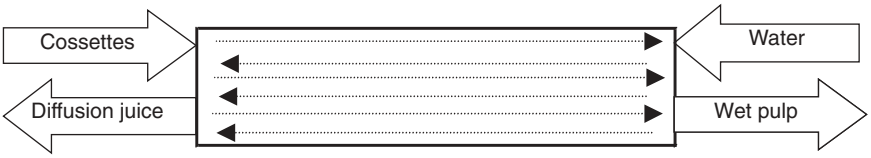


Figure 3.7.5 Countercurrent diffusion

In a continuous-countercurrent diffuser (see Figure 3.7.5), rotating equipment moves the cossettes continuously from one end, while water travels in the opposite direction. The cossettes gradually lose their sucrose (about 98%) and turn into **pulp** (desugared cossette), while the water gradually gains the sucrose and turns into diffusion juice, which usually contains about 15% *DS* (dry substance) with a **purity** (%sucrose in dry substance) of 85 to 88%. The rest of the sucrose (about 2%) stays with the pulp.

During diffusion, the juice is continuously pumped from the **head-end** (cossette side) of the diffuser, and the wet pulp is discharged from the opposite side, known as the **tail-end** (pulp side). The pulp is sent to pulp presses to be pressed. The pulp-press water from pulp presses is heated and returned to the diffuser as part of the diffuser water supply.

NOTES

- **Tower diffusers are equipped with a cossette mixer (scalder), which is a horizontal tank for preheating the cossettes. This type of diffuser results in a more efficient denaturation process. As the cossette enters the cossette mixer, it meets the warm juice. This brings the temperature of the cossettes to the denaturation level (about 70°C). The mixture of cossettes and juice is next pumped to the diffuser tower (the main part of the diffuser) for completion of the diffusion process.**
- **Despite the chemical changes in the cell's protoplasm caused by temperature, the cell wall (the main part of the pulp) retains its rigidity (hardness). Therefore, it continues to be resistant to mass transfer during the pressing process.**

FACTORS INFLUENCING THE DIFFUSION PROCESS

Factors influencing the diffusion operation are the following:

- pH
- Draft
- Temperature
- Retention time
- Cossette quality
- Microbiological activity

pH

Beet juice has a pH of 6.0 to 6.5. The optimum pH of diffusion juice is 5.8 to 6.0. At this pH, sucrose inversion to **invert sugar** (the 50:50 solution of glucose and fructose resulting from sucrose inversion) is at the lowest level. The acidification of diffusion water and sometimes pulp-press water is commonly practiced in the diffusion operation to slightly lower the juice pH and achieve the optimum value to prevent microbiological activity. Acidification also helps in the pulp-pressing operation.

Draft

Draft (refers to diffusion draft) is the mass of diffusion juice leaving the diffuser relative to the mass of cossettes entering the diffuser. It is expressed as percent on beet (% OB). For example, a draft of 120% means that 120 t of juice is produced from 100 t of processed beets. Draft not only indicates the amount of the diffusion juice but also helps determine the amount of **diffusion water** entering the diffuser (see the Material Balance of the Diffusion Process subsection in this section). Running the diffuser at optimum draft has the following benefits:

- High diffusion efficiency and low sugar loss in pulp
- Easy movement of cossettes and juice in the diffuser

Low draft slows the movement of the cossette-juice mixture and increases the risk of plugging the diffuser. High draft means extra water has been added to the process and will need to be evaporated later during evaporation. Today, a draft of 110 to 130 is considered an optimum range (depending on the type of the diffuser) when processing undamaged beets.

NOTE

The sugar factory has a weighing scale for measuring the mass of cossettes but usually has no scale for weighing diffusion juice or diffusion water. Therefore, the draft is calculated from known laboratory data. For calculating draft, refer to the upcoming Material Balance of the Diffusion Process subsection.

Temperature

Temperature (*T*) affects the diffusion process in the following ways:

- **Diffusion rate:** The higher the *T*, the better is the diffusion (the diffusion rate is three times larger at 70°C than at 20°C)
- **Denaturation:** The higher the *T*, the better is the denaturation (optimum *T* is 70°C)
- **Bacterial activity:** The higher the *T*, the lower is the level of bacterial activity
- **Cells behavior:** The higher the *T*, the more pectin is released to the juice (disadvantage)
- **Pulp-pressing ability:** The higher the *T*, the worse is the pulp pressability (disadvantage)

(Note: In the last two points, high temperature is a limiting factor.) Running the diffuser above 73°C causes a lot of damage:

- **Rupturing beet cells:** Ruptured beet cells allow the diffusion of more nonsugars (decrease in diffusion-juice purity) and pass part of the **pectin** into the juice
- **Swelling cossettes:** Swelling softens the cossettes because it loosens the binding between cells, which is secured by pectin (an insoluble polysaccharide)
- **Operational difficulties:** Swelled cossettes stick to the screen of the diffuser, slowing the movement of cossettes and plugging the diffuser
- **Pulp-pressing difficulties:** Pressing swelled pulp creates operational difficulties

On the other hand, running the diffuser at a temperature lower than 70°C causes the following problems:

- **Increased sugar losses:** Lower temperatures create an optimum medium for bacterial activities, causing sucrose inversion (sugar loss)
- **Operational difficulties:** Bacterial growth creates operational difficulties such as filtration difficulties

In view of all the pros and cons, the optimum temperature for the diffusion operation of undamaged beets is 70 to 73°C.

Retention Time

Retention time of the diffusion process is the amount of time cossettes are in contact with beet juice. A complete diffusion process takes place over a certain period. **Diffusion efficiency** (the degree of desugaring of the cossettes or nonsugar elimination) increases directly with time, meaning more sugar is transferred to the juice and less remains in the pulp. Retention time above the optimum creates some difficulties, such as the following:

- Decomposition of beet marc, slowing pulp-pressing operation
- Production of pulp with a high moisture content
- Increased risk of bacterial content

Differences in the beet quality and diffuser types make it almost impossible to recommend specific retention times for the diffusion process. The retention time, used by diffuser manufacturers for undamaged beet, ranges from 60 to 110 minutes.

Cossette Quality

High-quality cossettes are necessary to produce good diffusion juice with high purity without regard to the type of diffuser. See the slicing station (preceding section) for information about the quality of cossettes.

Microbial Activity

Cossettes are a good medium for the growth of many kinds of **microbes**, particularly **thermophilic bacteria** (grows around 50°C). This type of bacteria converts sugars to acids (mainly lactic acid), which aids in pulp pressing but increases sugar losses. In addition, it increases the soluble nonsugar content of the diffusion juice, which increases the amount of lime used in juice purification and juice softening (Chapter 6).

Microbes come with the cossettes to the diffuser where they reproduce particularly well when conditions are favorable for their growth (up to 20 million microbes can be found in 1 mL of diffusion juice.) At suitable conditions, microbes reproduce rapidly, and they can seriously contaminate diffusion juice. The considerable amounts of sugar decomposed in the diffuser provide food for their growth. More microbes are in the tail-end (pulp-side) of the diffuser than the head-end (cossette-side) for the following two reasons:

- Microbial activity is higher in the area where press water returns to the diffuser
- Microbial activity is higher at lower temperatures (the pulp-side has a lower temperature than the cossette side)

The presence of a high amount of mesophilic microbes such as *Leuconostoc* (a thermophilic microorganism that decomposes sucrose, resulting in the formation of lactic acid) in the diffuser is an indication of serious contamination.

Carefully washing the beets, feeding the diffuser with clean cossettes, and using clean diffusion water are important in diffusion operations. Temperature and pH control are other important ways to prevent the growth of microbes. As a rule, the lowest microbial activity is observed when the diffuser is kept at 70 to 73°C and at a pH of 5.8 to 6.0.

The practice of returning **pulp-press water** to the diffuser is another concern for contamination of the diffuser. Therefore, pulp-press water is usually heated before it is returned to the diffuser. The best way is to heat the press water to 90°C for several minutes, cool it to a temperature slightly lower than that in the diffuser (about 65°C), and then pump it to the diffuser.

DIFFUSION WATER

Supplying the diffuser with water of high quality can optimize the diffusion operation. Usually, **condenser water** (a mixture of condensate and cooling water) and **condensate** (water produced by the condensation of steam or vapor in the evaporation process) are used for the diffuser. The condensate in sugar factories is alkaline because of the ammonia content. Therefore, the pH can be reduced to about 5.6 by chemicals such as liquid sulfur dioxide (used mostly in North American beet-sugar factories), hydrochloric acid, or sulfuric acid (used mostly in European factories). Diffusion water with a high pH (above 7.0) will dissolve the pulp's pectin, which has the following disadvantages:

- Reduces the purity of the diffusion juice
- Reduces the amount of pulp that can be pressed because of pulp softening
- Reduces filterability of the juice during the next steps of the operation

ADDING PRESS WATER TO THE DIFFUSER

The wet pulp discharged from the diffuser is first pressed in the pulp presses to reduce pulp moisture (from about 90 to 75%). The press water from the pulp presses is screened to remove fine pulp particles, heated to 70 to 90°C, and pumped back into the diffuser. As noted, it is best to cool the press water to about 65°C before reusing it in the diffuser.

The heating of press water before returning the water to the diffuser **disinfects** the water to some degree, thus preventing sugar loss due to lower microbial activity. In addition, press water in some factories is treated with a **biocide** (a chemical for destroying microorganisms) on the way to the diffuser. The amount of pulp-press water is about 75 to 85% OB. The balance of the draft is fresh water. The press water is added near the pulp side of the diffuser where the juice has almost the same sugar content as the press water (1 to 3% sugar).

For a constant draft, returning press water to the diffuser decreases fresh-water requirement to the diffuser, so the sugar concentration in the diffusion water is increased. This decreases diffusion efficiency because the increase in sugar content decreases the concentration difference between the liquid phase within the cossettes and the diffusion juice around the cossettes, causing less diffusion efficiency (see the Notes below). Therefore, there is a limit for efficient pulp pressing because *the better the pulp pressing, the greater is the press water production*, which must be returned to the diffuser. Besides, the press water has a lower purity (about 70%) than that of diffusion juice, causing a reduction in juice purity.

NOTES

- **Returning press water to the diffuser is more of an environmental benefit (less load to the wastewater system) than a sugar-recovery benefit. However, this is a necessary operation in sugarbeet processing because of the energy cost of drying pulp and because it makes pulp handling easier. Steam-powered pulp dryers (used in modern beet-sugar factories) change that equation somewhat because they require less energy, but most factories are not equipped with this type of dryer. In addition, for a constant draft, less diffusion water is required because some of the water is replaced by press water.**
- **It is always better to aim for low pressed-pulp moisture because this can reduce the energy consumption necessary for drying the pulp.**

ADDING PRESSING AIDS TO THE DIFFUSER

Pressing aids (pressing agents) are chemicals added to the diffusion water to improve **pulp pressability** (the dewatering of plup by pulp presses). In some factories, pressing aids are added to the wet pulp going to the pulp presses or injected directly into the presses.

The pressing aid reacts with the pectin components of the pulp and raises the pulp-pressing efficiency. During the process, the cations of the chemical pressing aid react with the pectin to form pectate. The gelling increases pulp rigidity, which makes the pulp more suitable for pressing. Generally, adding a pressing aid to the diffuser has the following advantages and disadvantages:

- Increases amount of pulp (advantage)
- Increases pulp pressability (advantage)
- Decreases pulp moisture by 2 to 4% (advantage)
- Decreases the energy required for pulp drying (advantage)
- Decreases the need for juice-softening chemicals (advantage)
- Increases the natural alkalinity of the juice if alkali ions are displaced (advantage)
- Decreases the limesalts of the juice if alkali ions are displaced (advantage)
- Increases the load of nonsugars to the sugar-end (disadvantage)
- Increases carbonation-lime production (disadvantage)
- Increases molasses production (disadvantage)

In the sugar industry, the following chemicals are used as pressing aids:

- Gypsum ($CaSO_4.2H_2O$)
- Calcium sulfate ($CaSO_4$)
- Calcium chloride ($CaCl_2$)
- Aluminum chloride ($AlCl_3$)
- Aluminum sulfate [$Al_2(SO_4)_3$]

Use of 32% calcium chloride ($d = 1\,300\,kg/m^3$) in the amounts of 0.1 to 0.15% OB (1.0 to 1.5 kg/t beet or 2 to 3 lb/t beet) is common. Aluminum sulfate and calcium sulfate-dihydrate (gypsum) are also used in many sugar factories. Some sugar technologists suggest treating cossettes (before the diffusion process) with milk of lime, $Ca(OH)_2$, at about 0.15% CaO on beet.

Sugar factories equipped with the **juice-softening process** (see Chapter 6), in which calcium sulfate is the product of resin regeneration, use calcium sulfate as a pressing aid. In this type of juice softening, sulfuric acid regenerates the resin and calcium sulfate is the product of resin regeneration. The calcium sulfate solution usually has a dry substance of about 12%, which is stored and added to the diffuser as a pulp-pressing aid.

According to experiments at the Minn-Dak Farmers factory (Carlson et al. 1997), gypsum ($CaSO_4.2H_2O$) usage in the amount of 0.15% OB (1.5 kg/t beet or 3 lb/t beet) reduces pulp moisture considerably. This investigation also found that 65% of the sulfate stays in the diffusion juice, which increases the natural alkalinity and pH of the juice. The rest of the sulfate ends up in the pulp. On the other hand, almost all of the calcium ion goes with the pulp. As the following list indicates, for each 100 kg of gypsum used, the amount of pressed pulp increased by 16 kg and the ionic components of the diffusion juice increased by 56.4 kg:

SO_4^{2-}	33.2 kg
K^+	19.1 kg
Na^+	3.2 kg
Mg^{2+}	0.9 kg
Total	56.4 kg

The increase in juice cations causes an increase in molasses production, which is a disadvantage. The replacement of calcium ions (Ca^{2+}) for sodium and potassium ions during the diffusion process was found to be the reason for the increase in these ions in the juice. (Note: Na and K are highly melassigenic.)

ADDING ANTIFOAMING AGENT TO THE DIFFUSER

Foam (a colloid-type material, consisting of small bubbles of a gas trapped in a liquid) slows the movement of cossettes and juice in the diffuser, so an **antifoaming agent** is added to the diffuser to break up the foam molecules and improve the movement of the cossettes-juice mixture.

Antifoaming agents are added to in-process products (juices, syrups, and liquors) at different steps of sugarbeet processing to bring down the amount of foam (scum). The major cause of foaming in juice during sugar processing is the presence of **saponin**. Saponin solutions have low surface tension, which allows foam to form in in-process products.

Most antifoaming agents have a modified **fatty acid** as their main component. A few types of these agents are available to sugar factories. Usually, the required amount of antifoaming agent is about 100 g/t of beet (about 0.2 lb/t of beet).

ADDING BIOCIDE TO THE DIFFUSER

Bacterial activity can lead to considerable sugar loss during the diffusion process. The term **disinfection** refers to the microbe-destroying activity a **biocide**. Biocides and heat for reducing microbes should be used moderately because they can create side effects. For example, in the diffuser, high heat can kill most microbes but makes pulp pressing less efficient.

The use of a biocide during the diffusion process helps reduce microbial problems, prevents inversion of sucrose, and reduces acid (e.g., acetic, butyric, and lactic acids) formation. The sugar industry uses various biocides, such as formalin, sulfur dioxide, and chlorination (such as using hypochlorous acid, HOCl). (Note: The selection of biocide used in food industry and the amount used must meet legal standards.)

In the past, the most popular biocide was formalin (a 40% solution of formaldehyde). Continuous addition of formalin to the diffusion juice and press water at the rate of about 0.02% on beet prevents microbiological action in the diffuser. (But the application of formaldehyde has been discontinued in some countries and is expected to be discontinued in the remaining countries soon.) The discontinuation of formaldehyde is for one or more of the following reasons:

- Harmful to human health
- Affects natural environment
- Contributes to high limesalts
- Contributes to high ash in sugar
- Damages the respiratory and nervous systems
- Creates safety risks during handling and application
- Interferes with pulp pressing (decreases the *DS* of the pressed pulp)

Several attempts are being made by sugar technologists to find a suitable substitute for formaldehyde, including sulfur dioxide (SO_2). Today, sulfitation is used in many factories because sulfur dioxide is a good biocide, which improves sugarbeet processing in the following ways:

- Disinfects the diffusion juice
- Lowers the pH of the diffuser
- Improves the pressing qualities of the pulp because of the lower pH of the diffuser

- Reduces the color of the juice and also prevents color-formation in the next processing stations, where the temperature is too high (during evaporation)

Excessive use of sulfur dioxide, however, can lower the **natural alkalinity** (buffer) of the juice and increases thin juice hardness (disadvantages). Older factories produce sulfur dioxide in sulfur stoves. Most sugar factories now purchase liquid SO_2, which comes in a tank-truck and is loaded into a steel tank. The tank is installed outside the main building and the SO_2 is pumped through to the required stations. SO_2 is used at the rate of about 0.3 kg/t of beet (0.6 lb/t of beet) in the diffusion process.

Carbamates, ammonium compounds, and hydrogen peroxide (H_2O_2) are other biocides. These chemicals have been tested and sometimes are used by some factories to control microbial activities. Hop extract at 10 to 25 g per ton beets, applied in the upper part of the tower, have also been tested and shown to have reduced the lactic acid content of diffusion juice considerably (Hein 1997).

NOTE

A low level of lactic acid in the diffusion juice can be used as an indicator of reduced microbial activities. Since the 1990s, some sugar factories have measured the lactic acid in diffusion juice by a lactic acid analyzer that indirectly gives an estimate of the microbial activity of the diffuser. High levels of lactic acid (above 150 ppm) indicate bacterial growth in the diffuser because healthy beets contain very small amounts of lactic acid (namely L-lactic acid).

TYPES OF DIFFUSERS

Batch-type Robert diffusers (battery diffusers developed by Julius Robert in 1866) served the beet-sugar industry for more than 100 years until the late 1940s when **continuous diffusers** came on the market. Continuous diffusers can be divided into three main groups:

- Tower diffusers
- Slope diffusers
- Drum diffusers

Tower Diffusers

Tower diffusers have two main parts:

- Cossette mixer
- Tower

The **cossette mixer** (Figure 3.7.6) is used for preheating the cossette and for the denaturation of beet cells. The tower, the main part of the diffuser, is used for the actual diffusion process. The cossette first enters the cossette mixer, where it mixes with the warm diffusion juice, and then the mixture is pumped to the tower. A cossette mixer is more efficient when it is associated with a tower that can operate with thin cossettes having a Silin number of up to 13 with a low mush content. The two types of tower diffusers in common use are the BMA and Buckaw Wolf diffusers.

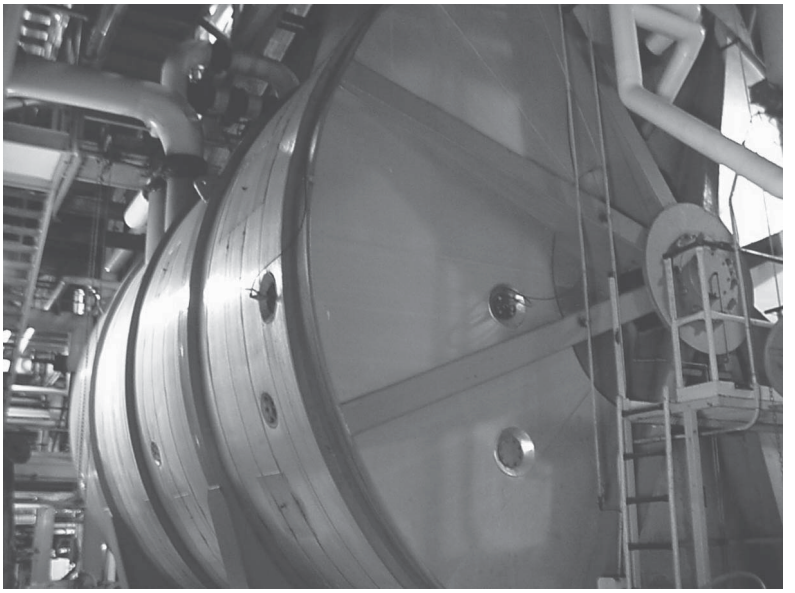


Figure 3.7.6 Cossette mixer

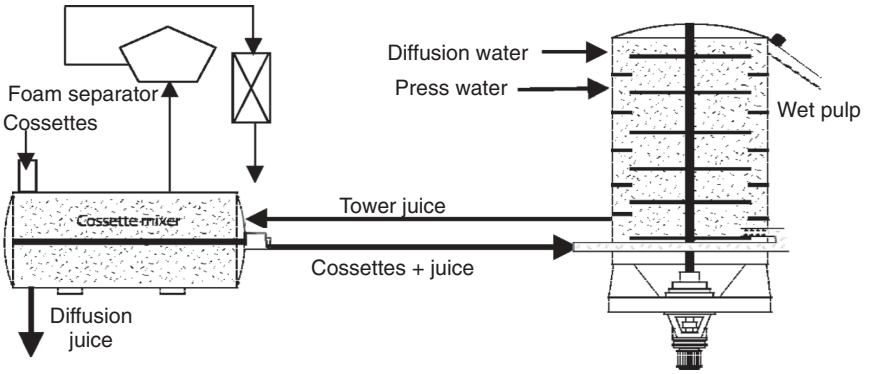


Figure 3.7.7 BMA diffuser

BMA Tower Diffuser

The **BMA tower diffuser** was developed by Braunschweig Machinenbau Anstalt (BMA) in Germany after the Second World War. A BMA diffuser 6.8 m in diameter and 23 m high can handle 5000 t beets/day. The newest model, which has a tower 11 m in diameter and 21.6 m high, can process 10000 t beets per day.

The cossette mixer of the BMA diffuser (Figure 3.7.7) is a horizontal cylinder with a perforated scroll that moves the cossettes forward at the desired rate. Juice from the bottom of the tower (called **tower juice**) is pumped (at a rate of 350 to 450% OB) into the cossette mixer. The

diffusion juice (equal to the required draft) is pumped from the opposite end of the mixer to the next step of the process, At discharge, the temperature of the diffusion juice is about 30°C.

The cossette mixer works as a heat exchanger because the hot tower juice preheats the cossettes. However, to keep the temperature of the cossette and juice leaving the mixer at approximately 65°C, part of the juice (about 80% OB) from the cossette mixer is pumped through a foam separator to a heater. The cossettes are heated further in the tower to a denaturation temperature (70°C) so that the sugar can be diffused out of the cells in the tower.

The mixture of cossettes and juice is pumped from the cossette mixer to the bottom of the tower. The **tower** is a 14 to 20 m vertical cylinder with a hollow shaft. In the tower, the cossettes are moved to the top of the tower by a rotating-lifting mechanism of several flights. The shaft rotates slowly, at 0.2 to 0.8 RPM (revolution per minute), and the flights are fitted on the shaft, giving the cossettes upward movement while the tower juice moves downward. Baffles are fixed to the walls of the tower to prevent the cossettes from rotating with the shaft.

Warm diffusion water (fresh water) is fed to the top of the tower. The heated **pulp-press water** is pumped back to the tower at a place where the sugar content of the juice is equal to that of the returned pulp-press water (the right place is about 20% from the top of the tower).

Diffusion water (fresh water) is added at the top of the tower, approximately 1 m (3 ft) below the pulp conveyor. The water moves through the moving cossettes. As the cossettes move to the top, more and more sugar is given up to the water, raising its sugar concentration. At the bottom of the tower, the tower juice is pumped to the mixer. Two screw conveyors discharge the **desugared beet cossettes** (wet pulp) from the top of the diffuser.

Buckaw Wolf Tower Diffuser

The **Buckaw Wolf** (BW) tower diffuser was also designed after the Second World War. The BW diffuser is similar to the BMA diffuser but with some differences in the cossette mixer and in the mechanism that lifts the cossettes inside the tower.

The cossette mixer of the BW diffuser is a horizontal cylinder consisting of scroll and mixer sections. The tower juice from the bottom of the tower is first passed through a sand catcher and then divided into two parts. The first part is pumped into the mixer section of the mixer. To heat the cossettes, the remaining juice (about one-third) and the circulating juice (pumped from the scroll section) are pumped to the heater and then into the scroll section. In the mixer section of the mixer, hot juice and fresh cold cossettes are mixed and then pumped back to the tower. The **diffusion juice** (equal to the required draft) is pumped out of the scroll section to the next step of the process.

In the tower, the water flows down, and the cossettes are moved to the top by a rotating-flight mechanism. The stationary baffles are mounted to the wall of the tower at a 22° angle. The wet pulp is discharged from the top of the tower.

Slope Diffusers

The **slope diffuser** consists of a horizontal drum 20 to 30 m (60 to 90 ft) long with a rotating mechanism for moving cossettes. The moving mechanism is either a chain type or a scroll type. Several slope diffusers are available, including the Silver-DDS and RT (Rotary Tirlemont) diffusers.

Silver-DDS Slope Diffuser

The **Silver-DDS slope diffuser** was originally developed by DDS. The first diffuser of this type with a capacity of 1200 t/d was installed in Denmark in 1952. Later, Silver Engineering modified the DDS diffuser and marketed it under the name of Silver-DDS diffuser. A few of these diffusers are in operation in the United States.

The Silver-DDS diffuser (Figure 3.7.8) consists of a U-shaped vessel, inclined at an angle of 11° to the horizontal. The cossettes move upward with the help of two rotating-parallel screws. The speed of the screws can be varied between 0.5 and 1.3 RPM depending on the slice rate. The wet pulp is discharged by double scrolls from the top-end (tail-end or pulp-end) where the diffusion water enters. The diffusion juice moves downward through the cossettes and, at the **low-end** (head-end or cossette side) of the diffuser, passes through a screen before it is pumped to the next step of the process. The screen separates the juice from the entering cossettes. There is no baffle or partition in this diffuser.

Steam jackets keep the temperature of the diffuser at around 70°C. The cossettes are heated at the low-end by the passing diffusion juice as the juice is cooled to about 25°C. Actually, the lower end of the DDS diffuser functions as the **cossette mixer** of the tower diffusers. The draft is regulated by addition of the diffusion supply water and by the slice rate.

NOTE

To increase diffuser efficiency and its throughput, some modern factories have modified their slope diffuser system by installing a countercurrent cossette mixer to fit their slope diffusers. This modification improves the thermal property of cossette before entering the diffuser. This type of modification (see Figure 3.7.9) raises the sugar recovered (decreases sugar losses) in the diffuser and its throughput. Since 1980, BMA has supplied several of such cossette mixers to Western European sugar factories.

Comparison of Tower and Slope Diffusers

Tower and slope diffusers have served the beet-sugar industry reliably for years, and the advantages of one over the other are not significant. Following are some comparisons of these two types of diffusers:

- **Simplicity of operation:** The operation of the slope diffuser is simpler because it does not include a cossette mixer, which can be somewhat complicated to operate.

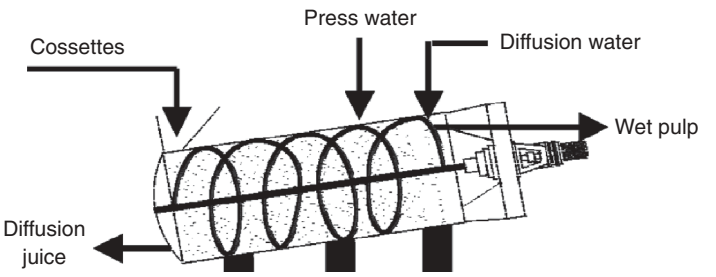


Figure 3.7.8 Silver-DDS diffuser

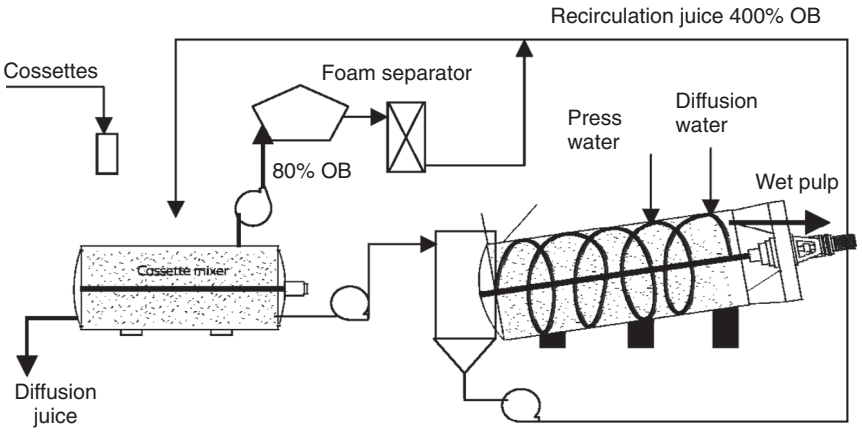


Figure 3.7.9 Modification of the slope diffuser

- **Silin number** (*SN*) **required:** Thinner cossettes with a high *SN* (greater than 10) result in better diffusion efficiency. But thin cossettes are damaged faster and turn into fine pulp, which can plug the screen and choke the diffuser. In this way, a slope diffuser has an advantage over the tower diffuser because it has no deflector (baffle); instead, it has screws and this feature causes less damage to the cossettes. Therefore, slope diffusers can handle thinner cossettes (larger *SN*) that have larger contact surfaces and thus provide better diffusion efficiency.
- **Intermixing:** Intermixing refers to the speed of movement of some cossettes compared to the other cossettes present in the diffuser. In both diffusers, intermixing of cossettes occurs because the cossettes near the inner shaft move more slowly than those farther out.
- **Energy consumption:** The slope diffuser consumes less steam than the tower diffuser. However, tower diffusers offer the opportunity for more waste-heat recovery and thus lower overall steam consumption.

PRODUCT AND BY-PRODUCT OF THE DIFFUSION PROCESS

Diffusion juice is the product and wet pulp is the by-product of the diffusion process. The diffusion juice has the following characteristics:

- A pH of about 6
- A dark-gray color
- A dry-substance content of about 15%
- Some sand and suspended solids (e.g., fine pulp particles)
- Some colloidal substances (e.g., pectin, and some coloring materials)
- Purity of 85 to 88%, depending on the quality of the beets and storage conditions

Before the diffusion juice is directed to the purification process (Station 10), it is screened to remove sand and pulp particles.

Wet pulp is the by-product of the diffusion process. Wet pulp has a high moisture content of about 90%. It is pressed to about 80% and dried to a moisture content of about 10%

before storage and transport. The dried pulp is valuable cattle feed, supplying carbohydrates, proteins, and minerals. Pulp pressing, drying, and pelleting are the subject of the next section (Station 8).

THEORY OF DIFFUSION

The **diffusion** of sucrose follows the general law of diffusion. What applies to the diffusion process applies to the crystallization (see Station 16) process as well. In the diffusion process, the molecules of sucrose and nonsucroses are the diffusing components; during crystallization, the *sucrose molecules* are the only component that is transferred from the syrup to the crystal structure. This is why the product of the diffusion process (diffusion juice) is not a pure sucrose solution (diffusion juice is 85 to 88% pure), but sugar crystals (after centrifuging) are 100% pure sucrose.

A. E. Fick originated the diffusion law in 1855. A little over one hundred years later, P. M. Silin reformulated the diffusion law for the diffusion process employed by the beet-sugar industry (Silin 1958). The Silin equation relates to the amount of the diffusing sucrose from the cossettes to the diffusion juice (C_3). Silin's law describes the concentration difference between sucrose in the cossettes coming to the diffuser (C_1) and going out with the pulp (C_2) (see Figure 3.7.10).

$$C_1 + C_4 = C_2 + C_3 \tag{3.7.2}$$

Because $C_4 = 0$, the sucrose concentration in the diffusion juice (C_3) is

$$C_1 - C_2 = C_3 \tag{3.7.3}$$

The amount of sucrose diffusing from cossettes with surface area of A and average thickness of d to the diffusion juice per time unit (t), which equals the concentration difference between cossettes and pulp, is given as

$$C_1 - C_2 = K_D \frac{A}{d} \times t \tag{3.7.4}$$

The K_D is the diffusion coefficient (the overall mass-transfer coefficient), defined as the rate of mass transfer per unit area per unit concentration difference. The concentrations can be expressed in percentage of mass (g/100 g water) or as mole fractions.

Albert Einstein formulated K_D in 1905 as

$$K_D = \frac{KT}{\eta} \tag{3.7.5}$$

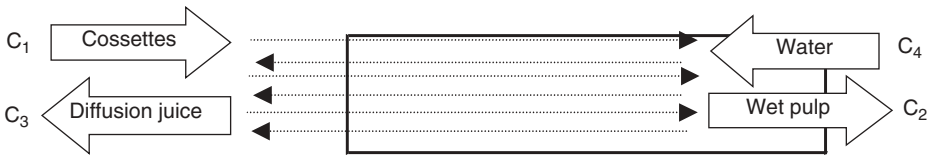


Figure 3.7.10 Diffuser inputs and outputs

Substituting K_D from equation (3.7.5) into (3.7.4) obtains

$$C_1 - C_2 = \frac{KT}{\eta} \times \frac{A}{d} \times t \tag{3.7.6}$$

K	Constant, dependent on the molecular mass of the dissolved substance
T	Absolute temperature (°C + 273)
η	Viscosity of the diffusion juice
A	Area of the cossettes
d	Thickness of the cossettes
t	Time (post denaturation time)

Silin considered the value of A/d as the length (in m) of 100 g cossettes; this was later called the **Silin number** (*SN*). The first point that we learn from the diffusion equation is that *the diffusion direction is from the higher concentration region inside the cells to the lower concentration region of water (or juice) around the cells*. In addition, the diffusion equation helps us find the factors that affect the diffusion process in order to optimize the diffusion process. Referring to the equation, we can state that diffusion:

- Improves with increasing time (t)
- Improves with increasing temperature (T)
- Improves with increasing cossette surface area (A)
- Worsens with increasing cossette thickness (d)
- Worsens with increasing juice viscosity (η)

K_D expresses the relationship between the diffusion process, temperature, the viscosity of the juice, and the constant K. Silin and Fo Schneider experimentally found a valid value for K equal to 6.5×10^{-5} (Silin 1958).

The K depends on the molecular mass of the substance to be diffused. *The larger the molecule, the smaller is K and the lower is the diffusion rate*. Proteins with high-molecular mass are diffused more slowly than sucrose, which is why a considerable amount of protein remains in the pulp. On the other hand, some nonsucrose substances diffuse more quickly than sucrose; therefore, this nonsucrose passes easily to the diffusion juice. For example, KCl diffuses almost five times more quickly than sucrose. The following table gives the diffusion coefficient (K_D), in cm^2/24 hours, of a few substances existing in beet juice at 70°C (Silin 1958):

Substance	K_D at 70°C
KCl	4.98
NaCl	3.88
K_2SO_4	3.04
Na_2SO_4	2.58
Sucrose	1.07
Raffinose	0.93
Proteins	0.03

DIFFUSION OF DAMAGED BEETS

Damaged beets (known as **Z-beets** to some sugar technologists) refer to beets damaged by unfavorable storage conditions or diseases. During the storage period, when the temperature changes from extremely cold to warm, frozen sugarbeets thaw and their cell walls become destructed (denatured), causing exposure to bacteria and thus damage to the beets. Beet damage increases when the weather change repeats two or more times. A similar problem can occur with beets stored in very hot weather. If these beets remain unprocessed for more than a week, they become badly damaged by microorganisms, especially the **mesophilic** types (mainly *Leuconostoc* bacteria), which gradually consume the sugar and convert some of it into **dextran** (see the following Note). At the same time, some of the sucrose hydrolyzes to **invert sugar** (a 50:50 solution of glucose and fructose resulting from sucrose inversion) and further to **lactic acid** and other acids. Dextran and levan are **colloidal substances** (colloids). Colloids include high-molecular substances such as pectin, dextran, coloring materials, decaying beet particles, and microorganisms. When the colloid content exceeds a certain amount, severe beet damage results. Damaged beets (see Figure 3.7.11) create operational difficulties mainly in the diffusion and filtration process.

In healthy beets, the dextran content is below 80 ppm. When the level exceeds 200 ppm, the beets become seriously damaged. Z-beets get soft; the outside color turns black; and the inside color turns orange. The diffusion juice produced from Z-beets is darker than normal juice. As a result, the juice's viscosity increases, causing serious difficulties in the process, particularly, in the filtration and settling operations. This brings the slicing rate down considerably (in serious situations, to about half the usual rate).



Figure 3.7.11 Damaged beets

Following are some recommendations for processing Z-beets during diffusion operations:

- Add more bactericide to decrease microbial activity and sucrose inversion
- Cut coarser cossettes because deteriorated beets produce mushy cossettes
- Run the diffuser at a higher draft to prevent high sugar loss in the pulp
- Add more antifoaming agent to the diffuser to prevent extra foaming
- Run the diffuser at a lower temperatures than usual (below 70°C)
- Add **dextranase** to the diffuser when beets are badly damaged

NOTES

- **Dextran is a long-chain gelatinous, water-soluble polysaccharide made up of glucose units. In beets, it is produced by *Leuconostoc* bacteria. Dextran has a high molecular mass.**
- **When the dextran content is high, difficulties occur in the diffusion and filtration process. The difficulties extend to the crystallization process, increasing the crystallization rate and creating elongated crystals. This is evident particularly in low-raw crystallization.**
- **Dextran affects polarimetric readings in laboratory tests.**
- **Dextranase is a biocide that breaks down dextran chains into smaller molecules. It is used at 5 to 8 ppm on diffusion juice (depending on the dextran content). Dextranase exhibits maximum activities at a pH of 5.0 to 6.0, and temperatures of 50 to 60°C. It can be utilized up to 8.0 pH and 80°C in the presence of high levels of sucrose. The best results are achieved when dextranase is added to the diffuser (low pH media) with press water or with cossettes on the cossette belt.**

MATERIAL BALANCE OF THE DIFFUSION PROCESS

In the **diffusion operation**, the quantitative control of mass transfer is based on the material balance. The following symbols are used in material-balance calculations:

M	Mass in kg (or in any other mass unit); for example, M_C = mass of cossette
W_S	Sucrose content in 100 kg of dry substance or liquid (% on sucrose); for example, $W_{S,PP}$ = % sucrose (*S*) in pressed pulp (*PP*)
W_{DS}	Dry substance content; for example, $W_{DS,Dif.J}$ = % dry substance (*DS*) in diffusion juice (*Dif. J*)
W_{NS}	Nonsucrose content, $W_{NS} = W_{DS} - W_S$

Draft

The **draft** (diffuser draft, *D*) is equivalent to the mass of the diffusion juice leaving the diffuser ($M_{Dif.J}$) divided by the mass of cossettes entering the diffuser (M_C), expressed as percent on beet (% OB):

$$D = \frac{M_{Dif.J}}{M_C} \times 100 \tag{3.7.7}$$

The sugar factory has a **weighing scale** for measuring the mass of cossettes, but usually no scale is used for weighing diffusion juice or diffusion supply water. Therefore, the draft is calculated from known laboratory data. For the calculation, the sugar content of the cossette

($W_{S,C}$), the sugar content of the diffusion juice ($W_{S,Dif.J}$), and the sugar loss in pressed pulp ($M_{Sl,PP}$) are used:

$$W_{S,C} = M_{Dif.J} \times W_{S,Dif.J} + M_{Sl,PP} \tag{3.7.8}$$

$$W_{S,C} - M_{Sl,PP} = M_{Dif.J} \times W_{S,Dif.J} \tag{3.7.9}$$

$$D = M_{Dif.J} = \frac{(W_{S,C} - M_{Sl,PP})}{W_{S,Dif.J}} \times 100\%\ \text{OB} \tag{3.7.10}$$

EXAMPLE 2

Given:

Sugar content of cossette, $W_{S,C}$	17.0%
Sugar content of diffusion juice, $W_{S,Dif.J}$	14.0%
Sugar loss in pressed pulp, $M_{Sl,PP}$	0.44% OB

Calculate:
Diffusion draft (D)

$$D = \frac{(W_{S,C} - M_{Sl,PP})}{W_{S,Dif.J}} \times 100 = \frac{(17.0 - 0.44)}{14.0} \times 100 = 118.3\%\ \text{OB}$$

Mass of Diffusion Water

The total masses entering and leaving the diffuser are equal:

$$M_{Cossette} + M_{Dif.W} = M_{Dif.J.} + M_{\text{Pr.}pulp} \tag{3.7.11}$$

or

$$M_C + M_{Dif.w} = M_{Dif.J} + M_{PP} \tag{3.7.12}$$

$$100 + M_{Dif.w} = M_{Dif.J.} + M_{PP} \tag{3.7.13}$$

$$M_{Dif.w} = M_{Dif.J} + M_{PP} - 100 \tag{3.7.14}$$

(Note: In the preceding equations, the mass of the **pulp-press water** is not included, since it returns to the diffuser after the pulp is pressed.)

EXAMPLE 3

Given:

Mass of cossette, M_C	100 kg
Mass of diffusion juice (draft, D), $M_{Dif.J}$	111% OB
Mass of pressed pulp, M_{PP}	18.4% OB

Calculate:
Mass of diffusion water on beet (% OB)

$$M_{Dif.w} = M_{Dif.J} + M_{PP} - 100 = 111 + 18.4 - 100 = 29.4\%\ \text{OB}$$

OPERATING PROBLEMS

A number of **problems** can occur in diffusion operations: high sugar loss in pulp, low diffusion efficiency, low diffusion-juice concentration, and low diffusion-juice purity.

High Sugar Loss in Pulp

The following are the usual causes of high sugar loss in pulp:

- **Low draft:** Draft (diffuser draft) is the most important factor affecting sugar loss in pulp and diffusion efficiency. Decreasing the draft by 1% increases the loss by almost 0.02% on beet (OB). A lower draft gives lower rates of diffusion and typically higher sugar loss in pulp (lower diffusion recovery), but it requires less energy for evaporation. Today, the beet-sugar industry uses a draft between 110 to 120% as the optimum range.
- **Wrong cossette size:** Cossette size, in particular, its thickness, affects the sugar loss in pulp and the movement of cossettes in the diffuser. Thin cossettes produce pulp with less sugar content and higher diffusion efficiency. However, thin cossettes move in the diffuser with difficulty and can plug the diffuser. Therefore, there should be a compromise between cossette thickness and sugar loss. On average, 4 mm thick cossettes are optimum when healthy beets are processed. Cossette height is judged by the *SN* (Silin number). When healthy beets are processed, the higher range of *SN* should be used to accomplish diffusion with maximum efficiency. Some factories use a coarser knife size (8.2 × 6 mm) when damaged beets are processed to prevent movement difficulties and plugging during diffusion. In this case, a higher draft is recommended to prevent high sugar loss to pulp. All diffusers require a maximum mush content of 5%. The DDS diffuser can operate with thinner cossettes (higher *SN*).
- **Wrong temperature:** In general, the diffuser has better efficiency when it operates at about 73°C. At high temperatures (above 73°C), the cell wall structure is increasingly broken down into small, degraded particles of protein. These particles can end up in the diffusion juice, where they cause lower diffusion-juice purity, foaming, slow settling, and filtration problems. In addition, operating the diffuser at high temperatures decreases its pulp-pressing ability. In the BMA diffuser, the amount of the circulation juice for heating is about 300% OB. If additional heating is necessary, a portion of the tower juice can be heated before it enters the cossette mixer to keep the content of the cossette mixer at about 70°C.
- **Short retention time:** Cossettes are retained in the diffuser for a longer time than the juice because the juice moves faster. The design of the diffuser and the method of cossette movement in the diffuser also affect retention time. Remember that the diffusion time should be considered as postdenaturation, which is the period beginning the moment that the temperature of the cossettes reaches 70°C. Usually, it takes about 8 minutes at 70°C to denature (kill) the protoplasm of the cell. In general, if other conditions are kept constant, more time will enable a complete diffusion process. High retention time recovers more nonsucroses and increases the risk of bacterial growth.
- **High pH:** The natural pH of beet juice is about 6.0. The optimum pH for diffusion operations is about 6.2. Running the diffuser at a high pH (above 6.5) causes the cossettes to soften, resulting in greater sugar loss in pulp. Over 6.5 pH, the cell wall pectin becomes soluble, which increases impurities in the diffusion juice and leads to mushy cossettes, poor pulp-pressing operations, more foaming, and potential filtration problems. Measuring the

pH at regular intervals (at least two to three times a shift) can keep the pH under control. A pH drop below 6 in the center of the diffuser indicates microbial development and calls for immediate preventive measures against sugar losses. In addition, at a low pH, the rate of equipment corrosion is greatly increased. The diffusion-supply water is kept between 5.5 and 5.8 pH by using sulfur dioxide (SO_2).

- **High sugar content in beets:** Sugar loss in pulp is directly proportional to the sugar content of the beet. In other words, under the same processing conditions, beets with higher sugar content will have higher sugar loss in the pulp unless the draft is increased.
- **Low feed rate:** Usually at the beginning and toward the end of the campaign and during slow operations, the factory needs to feed the diffuser partially. This causes a problem with the movement of cossettes in the diffuser. The decrease in the speed of the shaft increases the speed of the cossettes, which can cause more sugar loss to pulp because the retention time shortens. In this situation, thinner-sliced cossettes are recommended because they move more slowly in the diffuser than thicker cossettes.
- **High microbial growth:** Despite washing, cossettes entering the diffuser will contain some microbes, which can cause sucrose inversion and sugar loss. Careful washing of beets and clean pulp-press water are important ways to reduce microbial activity in the diffuser. To fight microbes, the pulp-press water is usually heated before it is returned back to the microbes. The best method is to heat the press water to 90°C for several minutes to complete the pasteurization, followed by cooling it to a temperature slightly lower than that of the diffuser (about 65°C), or by heating the water to 70°C and holding it at that temperature for 10 to 15 minutes. Usually, lower microbial growth is observed when the diffuser is kept at 65 to 70°C, the pH is 5.8 to 6.0, and the diffuser is fed with clean and steam-treated cossettes. All losses caused by the previous factors are considered known losses. Sugar loss caused by microorganisms is called an **unknown loss**.

Low Diffusion Efficiency

Following are the usual causes of low diffusion efficiency:

- **Low-quality cossette:** Low-quality cossettes are damaged more quickly, resulting in a plugged diffuser. As mentioned, the thickness of cossettes, the Silin number, and the cossette mush content are the most important quality factors of the cossette.
- **High temperature:** Overheated cossettes (above 73°C) are softer and less resilient, move more slowly, and become swollen (expanded). Swollen cossettes easily plug the screen of the diffuser, causing lower draw off of the diffusion juice.
- **Low draft:** Low draft plugs the screen and can choke the diffuser. Low draft also affects the purity of the diffusion juice.
- **Low marc content:** The **marc** (the water-insoluble component of sugarbeet) content of the beet decreases during storage. As a result, the cossette softens, which makes the extraction of sucrose less effective.

Low Diffusion-Juice Purity

A diffusion juice with purity of about 88% is expected from processing normal beets. A diffusion juice produced from normal beets with low purity is an indication of a malfunction.

If the diffusion juice produced under correct processing conditions is of low purity, the cause might be low-quality beet with regard to the beet storage or chemical properties. Climate can affect the quality of the beet considerably. During storage, reactions take place on most of the beet substances, such as sucrose, amino acids, and pectin (partially water-soluble, gel-type, made of galactose units), which lessen the quality of the beet, resulting in low diffusion-juice purity. The following are a few causes of low diffusion-juice purity:

- **High nitrogen in beets:** When beets do not get enough water during the growing period, and are overfertilized with nitrogen, the amount of organic nonsugars, particularly amino acids, increases. Usually, a low-quality diffusion juice is produced when beets with high-nitrogen content are processed.
- **High invert sugar in beets:** Unripe beets and beets stored for a long period under unfavorable conditions have a high invert-sugar content. These types of beets usually produce low-quality juice.
- **Damaged beets:** Damaged beets (**Z-beets**) refer to beets damaged by storage or disease. Reasons for beets being damaged by storing are the following:

 High temperature

 High dirt content

 Freezing and thawing

The amounts of invert sugar, dextran, and levan in damaged beets are greater than usual. This creates difficulties during diffusion and filtration of the juice in further operating steps.

CHAPTER 3

SECTION 8

PULP TREATMENT

SUBSECTIONS

- Pulp Pressing
- Pulp Drying
- Pulp Pelleting
- Pulp Storage
- Pulp Composition and Usage
- Material Balance
- Operating Problems

ABOUT THIS SECTION **This section** explains **Station 8** (Pulp Treatment) of sugarbeet processing. Station 8 (see Figure 3.8.1) consists of two steps: pulp pressing and pulp drying. In some factories, a third step of operation is also employed: pulp pelleting. When the exhausted (desugared) beet cossettes leave the diffuser, they are called **wet pulp**, containing about 90% water (10% dry substance [*DS*]) and almost all the **beet pulp** (beet's fibrous materials). The wet pulp still contains a considerable amount of sugar (sucrose). To recover the sugar from the wet pulp and handle, store, and market the wet pulp economically, it must be pressed and dried to decrease its moisture content to about 10% (90% *DS*). This is achieved in a two-step process. First, the wet pulp is pressed in **pulp presses** to about 75% moisture content (25% *DS*). The juice pressed from the pulp, called **press water**, is screened, heated, and returned to the diffuser as part of the diffusion water. In the second step, the **pressed pulp** is dried in **pulp dryers** to about 10% moisture content (90% *DS*).

In factories with pulp-pelleting equipment, the dry pulp is pelletized (sometimes with the addition of molasses) in **pellet presses** (pelletizers) to produce **pelleted pulp** (pellets). Pelletizing puts the pulp in a compact form that can be economically transported to pulp users (mainly the animal feed industry).

Beet-Sugar Handbook, by Mosen Asadi
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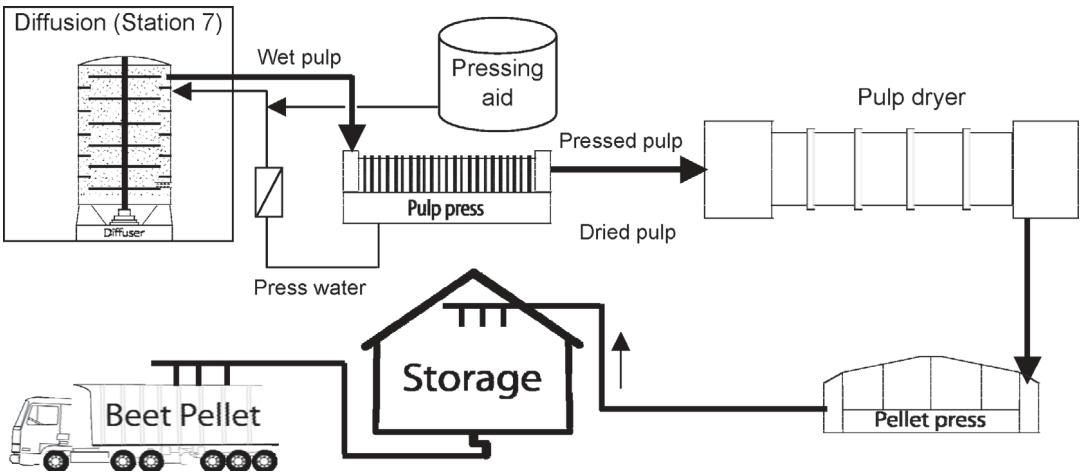


Figure 3.8.1 Pulp-treatment operation (Station 8)

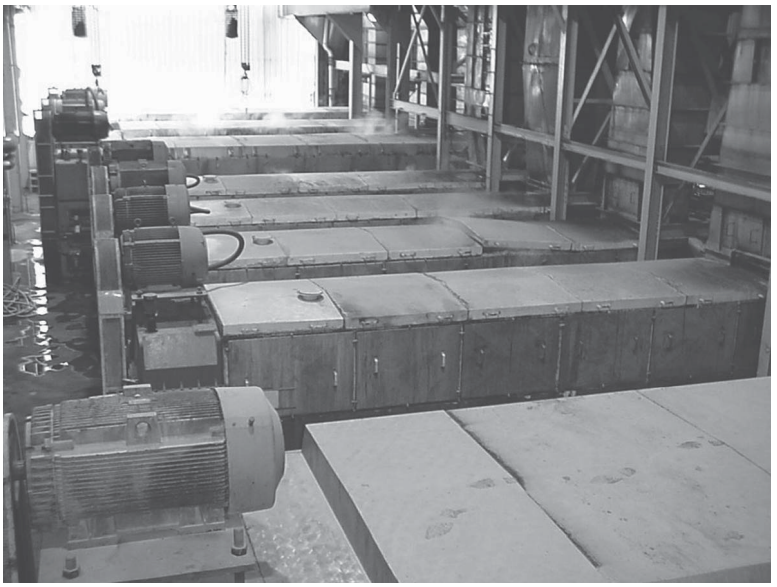


Figure 3.8.2 Pulp presses

PULP PRESSING

The **wet pulp** leaving the diffuser is transported by a screw conveyor to the pulp-pressing section. In the factories equipped with the **chip separator** (see Section 5 of this chapter), trash particles separated from the flume water are also sent to the pulp-pressing section to be pressed along with pulp. In the pulp presses (see Figure 3.8.2), the wet pulp is pressed to a smaller

volume with about 75% moisture content (25% *DS*). (With the use of a pressing aid, pulp pressability improves, resulting in moisture content reduction to about 65%.) The press water is collected in a tank and then pumped to a screen, where fine pulp particles are removed and returned to the pulp presses for repressing. The screened press water, called **pulp press water**, is then heated to 70 to 90°C before being returned to the diffuser. The amount of pulp-press water is about 75 to 85% OB (on beet). Heating disinfects the press water before it is returned to the diffuser, preventing sugar losses due to microbial activity. In addition, the press water in some factories is disinfected with a **biocide** (a chemical for destroying microorganisms) on its way to the diffuser.

It is generally cheaper to remove water from the wet pulp mechanically by pressing than by thermally drying. The water content of the wet pulp can be reduced by the presses as much as practicable before feeding the pressed pulp to the pulp dryer. The pulp-pressing operation is important because it

- Removes about 80% of the total water present in the wet pulp
- Provides part of the needed water for the diffusion process
- Reduces the load to the wastewater system
- Saves energy in pulp drying

Without pressing, it would be uneconomical to dry the pulp because doing so would require too much energy. (Pulp pressing removes about two-thirds of the water in the wet pulp.)

The following factors affect **pulp pressability** (the degree of dewatering of the pulp by pulp presses):

- Pressing aids
- Size of cossettes
- Beet marc (fiber) content
- Physical properties (rigidity and degree of damage) of the beets
- Diffuser operating conditions (temperature, draft, retention time, and pH)

Pressing aids (pressing agents) are the chemicals added to the diffuser to improve pulp pressability. Pressing aids can be added to the diffusion water going to the diffuser, to the wet pulp going to the presses, or sometimes directly into the presses. BMA showed (Bosse 1997) that injecting a gypsum **slurry** (suspension) directly into the pulp presses (BMA vertical-type presses) increases the *DS* content of pressed pulp considerably. In this system, the slurry is added to the lower part of the press where most of the press water has already been removed. This practice improves the pressing efficiency of the lower part of the presses.

The chemicals used as pressing aids contain calcium cations (Ca^{2+}). The calcium cations bind tightly with the pulp (particularly, with the pectin in the pulp), improving its rigidity and pressability. Because of the added pressing aid, calcium ions are exchanged for sodium ions (Na^{+}) of the juice during the diffusion process. Most of the released calcium of the juice goes with the pulp, resulting in a softening action to the beets (Ca is a major component of hardness).

In general, using pressing aids

- Coagulates colloids present in the pulp
- Decreases the amount of water to the dryer

- Increases pulp-pressability by making the pulp more rigid
- Decreases the amount of softening chemicals that has to be added to the process later

The disadvantage associated with pressing aids is that their ions increase the load of nonsugars in the juice, resulting in higher molasses production.

Section 7 of Chapter 3 described the diffusion operating conditions (temperature, draft, retention time, and pH) that affect pulp pressability. A diffuser operating at about 70°C creates the best condition for pulp pressing. By the time pulp reaches the presses, its temperature has dropped to about 60°C, which is optimum for pulp-pressing operations. Operating the diffuser at about 6.2 pH results in the best pressing operation. At a high pH (above 6.5), the cell wall pectin becomes soluble, producing softer pulp and decreasing pressing efficiency. Figure 3.8.3 shows the effect of pH on the pressing operation (van der Poel 1998).

Calcium sulfate-dihydrate (gypsum, $CaSO_4.2H_2O$), calcium sulfate ($CaSO_4$), and calcium chloride ($CaCl_2$) are the most frequently used pressing aids in the beet-sugar industry (see Station 7 for more information). Gypsum in the form of a 12% solution and at 0.15% OB (1.5 kg/t beet or 3 lb/t beet) improves the pressability by 5 to 10%. Theoretically, every kg of gypsum takes about 230 kg of water out of the pulp, but in practice, the figure is about 80 kg. There is a 15 kg increase in pulp production for every 100 kg of gypsum used. As a result, the amount of sulfate $(SO_4)^{2+}$ in the diffusion juice increases, resulting in increases of pH and the natural alkalinity of the juice. Figure 3.8.4 shows the relationship between the amounts of gypsum added and sulfates in the diffusion juice (Carlson et al. 1997).

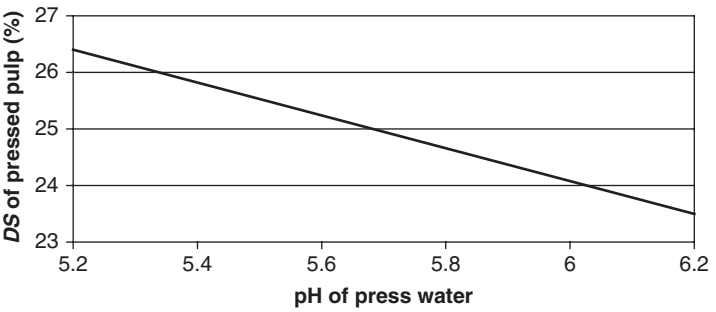


Figure 3.8.3 Relation between the pH of press water and the dry substance (*DS*) of pressed pulp

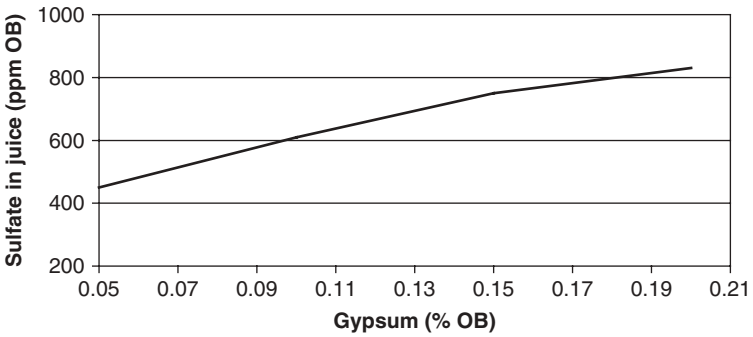


Figure 3.8.4 Relation between gypsum use and sulfate in the diffusion juice

Calcium sulfate ($CaSO_4$) can be produced on site by adding sulfuric acid to milk of lime (MOL).

$$H_2SO_4 + Ca(OH)_2 \rightarrow CaSO_4 + 2H_2O$$

The level of *DS* (dry substance) in the pressed pulp depends on both the rigidity of the pulp and the type of pulp press. Pulp presses are of two types:

- Vertical single-screw pulp press
- Horizontal twin-screw pulp press

NOTES

- **Some sugar factories use a light-pulp pressing to about 85% moisture content (15% *DS*), which saves on energy costs. Because of spoilage, light-pressed pulp should be used within 5 to 7 days of delivery.**
- **Some factories mix pressed pulp with molasses (at about 10% on pulp mass) and some minerals and sell it as pressed-molassed pulp.**
- **Because of today's increasing fuel costs, the production of pressed and ensiled pulp is expected to increase in comparison to dry pulp.**

PULP DRYING

Pressed pulp is directed by a belt conveyor to the pulp dryer to produce **dry pulp** with about 10% moisture content. Wet-pulp particles are highly porous, so moisture can be easily removed in the dryer, where heat is formed by the combustion of fuel with air to evaporate water from the pulp. In the feed-end of the dryer, no drying takes place until water reaches its evaporation temperature. This period is known as the **zero-rate period**. As pulp particles move farther, drying starts and continues over a longer period, known as the **fast-rate period**. During this period, the drying rate stays almost constant or decreases slightly, and the temperature of the drying fuel decreases while the temperature of the pulp increases. At the discharge end, both temperatures are the same, and the drying rate is at its lowest rate, hence known as the **slow-rate period**.

Coal, oil, or natural gas are used as the fuel in the pulp dryer. Beet factories usually use drum dryers, which have a furnace similar to the furnace in a boiler. Fuel oil furnaces have a burner for injecting the fuel. Solid-fuel furnaces have a bunker.

The oxygen needed for the combustion reaction is provided by air, which must be mixed with fuel in the most efficient way. The air we breathe is a mixture of nitrogen (N), oxygen (O) and water (its amount depends on the weather's moisture), and a small amount of carbon dioxide (CO_2). A typical **dry air** contains about 21% (by volume) of oxygen, 78% nitrogen, and 1% carbon dioxide gas.

The **combustion air requirement** (CAR) is kg (or lb) of air needed for combustion of each kg (or lb) of fuel. The CAR is calculated based on fuel analyses (see Section 9). Some excess oxygen above the theoretical amount is required to achieve a complete combustion of the fuel. Without sufficient oxygen, the combustion is incomplete and produces carbon

monoxide (CO), which is *hazardous*. An excess air requirement (EAR) coefficient of 1.3 is often used. Theoretically, to burn 1 kg of fuel oil, 13.4 kg of dry air are needed. Therefore, the practical need (by applying a coefficient of 1.3) is equal to 17.4 kg of dry air per kg fuel. Or, for combustion of each cubic meter of natural gas, 10.2 m^3 air is needed. A coefficient of 1.3 brings this amount to 13.3 m^3 of air.

Pulp dryers are of two types:

- Drum dryer
- Steam-powered dryer

Drum dryers are popular in the sugar industry because they can use natural gas, oil, or coal as fuel. A drum dryer is a large rotating drum that rotates at about 2 RPM (revolution per minute) to prevent the pulp from sticking to the drum wall. Drum dryers use forced draft and fuel to produce hot-dry air (700 to 900°C) to remove the water from the pulp. An average-size drum dryer is 4 m (12 ft) in diameter and 16 m (48 ft) long and can process pulp produced from 3000 t of beet a day. A larger unit is 5 m (15 ft) in diameter and 20 m (60 ft) long and can handle 5000 t of beets a day. The furnace of a drum dryer can use natural gas, oil, or coal as fuel. A forced draft for fuel combustion is created by fans, which are located at the discharge end of the drum to force air into the drum.

At the discharge end of the drum, the drying gas passes through an expansion vessel to separate the drying gas from the dry pulp. The exhaust gas then passes through the cyclone and enters the **dust scrubber** (wet dust collector) by force of a fan. From the scrubber, the exhaust gas is vented to the air by a chimney. (The exhaust gas leaving the chimney is about 120°C, which is considered heat lost.)

To save heat energy, exhaust-gas return to the dryer furnace is practiced in some factories. The return gas increases the **specific enthalpy** (the quantity of heat required to transform 1 kg of boiling water into 1 kg of saturated steam) of the combustion gas. Because of its high moisture content, the return gas increases the water content of the combustion gas, which is a disadvantage. This method can reduce fuel consumption by about 6% (van der Poel et al. 1998).

Steam dryers use **superheated steam** from the boiler house for pulp drying. The vapor removed from the pulp then serves as heat in the vapor users of the factory. Steam dryers do not demand changes in the boiler house. Today's increasing fuel costs often justify the replacement of drum dryers by more efficient steam dryers. Pulp drying by steam dryer provides the following advantages compared with fuel-burning dryers:

- Savings on energy cost
- Reduction in air pollution

In recent years, steam pulp dryers have been installed in several beet factories around the world. Steam dryers may become the standard method of pulp drying in the near future because of the reasonable **return on investment** (ROI) they provide. The Minn-Dak factory in the United States installed a steam pulp dryer built by Enerdry in 2003. Because the factory does not produce electric power, a steam turbine using 250 psig steam pressure was used. The project ROI was about three years (Thilmony et al. 2005).

NOTES

- **The pulp-dryer foreman is the person responsible for running the pulp-drying operation. The foreman's main duty is to regulate the fire and the feed inside the pulp dryer to ensure that the pulp has the desired moisture.**
- **Today, a moisture analyzer is usually placed in the control room of the pulp-drying station, so the pulp foreman can determine the pulp moisture content in five minutes. Moisture analyzers are equipped with a heating element and a microbalance. The microbalance automatically weighs the sample before and after drying, and calculates the percentage of moisture.**
- **Pulp drying is energy intensive (consuming about one-third of the total factory energy). The recent increase in energy costs has led the sugar industry to consider pulp-drying methods that demand less energy (e.g., drying with solar and wind energy). A sugar factory in California has been using solar and wind energy successfully since 1980 (van der Poel et al. 1998). In this factory, concentrated Steffen filtrate (CSF) from the Steffen molasses-desugaring process and later raffinate from molasses desugaring by chromatographic (MDC) process are mixed with the pressed pulp. The mixture is transported by a screw or belt conveyor to the drying area (0.5 km from the factory), and spread with a truck to a thickness of about 2 cm. The drying area is fenced to avoid pulp loss and is divided into lanes about 6 m wide. The ground is graded to a slope of about 1% for drainage. The soil is stabilized to a depth of 30 cm using lime (CaO). During the drying period, the pulp is turned when its surface becomes dry. The turning process is continued until the pulp reaches a water content of about 10%. In this process, wind speed plays a more important role than high temperature.**
- **Ensiling wet or pressed pulp is practiced in some countries (e.g., Denmark) to preserve it. Ensiled pulp is used on farms for feeding animals. More information about this process can be found in *Sugar Technology, Beet and Cane Sugar Manufacture* (van der Poel et al. 1998).**

PULP PELLETING

Today, most factories pellet (form into pellets) the dry pulp in **pellet presses** (pelletizers) to produce **pelleted pulp** (pellets). Some factories pellet the dry pulp with the addition of molasses.

The pellets are round, 6 to 16 mm (1/4 to 3/4 in.) in diameter and 25 to 50 mm (1 to 2 in.) in length. Pelleting puts the pulp in a compact form (the bulk density of pellets is about triple that of dry pulp), which creates the following advantages:

- Less storage area
- Lower risk of fire
- Lower handling expenses
- Less fine-particle formation during transport

The dry pulp from the dryer is supplied to a surge tank above the pellet presses. Then, the pulp is mixed with steam or hot water to increase its temperature to 90°C. For pelleting, dry pulp must have 8 to 12% moisture content. In some factories, molasses is also added (as explained later). The moist pulp is fed to the center of a rotating die in the pellet press, and then forced to the die openings by the rollers. Knives, positioned on the side of the die, cut the pellets to the desired length. The length can be changed by adjusting the clearance between the die and the roller.

Two types of pellet presses are usually used in the sugar industry:

- Ring-collar press
- Disk-collar press

The pelleting operation is intensive and causes considerable wear to the dies and rollers, particularly when the pulp contains sands.

Adding Molasses to the Pulp

In some factories, molasses is added to the pulp to produce **molassed pulp**, an animal feed with a high sugar content. Molasses is added in one of the following ways:

- To the pressed pulp before drying to produce **pressed-molassed pulp**
- To the dry pulp after drying to produce **pelleted-molassed pulp**

In the first method, the pressed pulp and molasses are mixed in a mixer with about 15 minutes retention time. Typically, 20 tons of molasses are mixed with 100 t of pressed pulp to produce 50 tons of dry pulp at 10% moisture content. In the second method, the dry pulp is mixed with molasses and steam (or hot water) in a mixer at about 90°C before pelleting. Diluted molasses works as a lubricant, reducing the wear on the die of the pellet presses (pelletizers).

If the molasses is added to the pressed pulp before the dryer, a more uniform product is produced (compared with postdrying addition of molasses) but with the following disadvantages:

- Increased odor during pulp drying
- Decreased nutritional value of the molasses components by combustion

The optimum amount of added molasses is about 10% on pulp mass, which produces molassed pulp containing about 10 to 11% sugar (5% from molasses and 5 to 6% from pulp). However, molasses can be added up to 30%, depending on the user's requirements (van der Poel et al. 1998). Molasses addition increases

- Income of the factory
- Amino acids of the pulp
- Sugar content of the pulp
- Handling quality of the pulp

NOTE

In factories equipped with molasses desugaring by chromatographic (MDC) process, raffinate (also called CMS for concentrated molasses solids) is added to the pulp instead of molasses. CMS is a by-product of the MDC process.

PULP STORAGE

Dry pulp leaving the pulp dryer is hot (60 to 90°C), so it is cooled in the **pulp cooler** and then stored in the **pulp storage** (pulp silo or pulp warehouse) until sold, mainly in bulk form. When a factory produces **pellets**, the cooling process is not required, and the pellets are moved directly to storage. The pulp storage is adjusted for temperature and humidity by ventilation and air circulation to maintain the moisture content of the stored product at a desired level. When the air moves through the pellets, it absorbs moisture and increases in temperature.

Inadequate air circulation and a cold climate cause moisture migration (due to the temperature difference between the top and bottom of the pile), resulting in a hardening of the pellets. In addition, there is the risk of mold formation.

Some modern factories store pellets in a silo similar to a sugar silo. To simplify calculations for transportation, the following table indicates the approximate **bulk density** of the pulp:

	Density (kg/m^3)	Density (lb/ft^3)
Pressed pulp	600–640	37–40
Dry pulp	200–240	12–15
Dry-molassed pulp	200–220	12–14
Pellets	640–720	40–45

NOTE

Because of fire risk, the amount of dust in the pulp warehouse must be controlled at all times by proper ventilation and airflow.

PULP COMPOSITION AND USAGE

Pressed pulp consists of mainly beet marc (about 5% on beet), proteins (about 0.5% OB, almost two-thirds of the beet proteins), and sugar (0.2% OB). When the wet pulp is pressed, part of its marc content (0.5% OB) and a small amount of its protein (0.1% OB) are lost to press water. **Beet marc** consists mainly of pectin, cellulose, and hemicellulose. Humans lack the enzyme that breaks up the cellulose and hemicellulose, and so cannot digest the beet pulp. Cattle, however, do have this enzyme, which converts cellulose and hemicellulose into small digestible molecules that can be used by the animal as a nutrient.

Beet pulp (in dry, pelleted, or ensiled form) is used as a high-energy, low-protein supplementary food for **ruminants** (animals that bring the food back from their stomach into their mouth for re-chewing, e.g., cows). It is used to feed cows, horses, and pigs. In terms of food value, pulp ranks between hay and oats. Its digestibility is 1.5 times higher than hay. The protein digestibility of the pulp is about 75%.

The dry pulp's protein content is slightly less than that of hay and oats. Its **metabolizable energy** (ME; the energy provided by a food source after digestion) is about 2700 cal/kg (corn has 3300 cal/kg). Compared with corn, beet pulp results in lower pH in animals after feeding. Following is the typical composition of dry beet pulp:

Dry Pulp Composition	
Marc	68.0% m/m
Sucrose	7.0%
Protein	7.0%
Ash	5.0%
Minerals	2.5%
Fat	0.5%
Moisture	10.0%

MATERIAL BALANCE

In **pulp operation**, the following material balances are important:

- Pulp yield from pulp pressing
- Pulp yield from pulp drying
- Sugar loss in the pressed pulp

Here, the calculations for mass balance of these materials and the energy balance of pulp drying are provided.

Wet Pulp, Pressed Pulp, and Dry Pulp Yields

The **pulp yield**, which is the amount of pulp produced per 100 kg of beet (or % OB), is calculated from the pulp content of cossettes (usually 5% OB in practical calculations). Marc (pulp) is the insoluble component of the beet that usually passes into the pulp without considerable change. Therefore, the marc content of cossettes ($W_{M,C}$), of wet pulp (W_{MWP}), and of pressed pulp ($W_{M,PP}$) is the same:

$$W_{MC} = W_{MWP} = W_{MPP} \tag{3.8.1}$$

The wet pulp yield (M_{WP}), which is the mass of wet pulp produced per 100 kg of beet, is calculated from the marc content of cossettes ($W_{M,C}$) and the % dry substance (DS) of wet pulp ($W_{DS,WP}$):

$$M_{WP} = \frac{W_{M,C}}{W_{DS,WP}} \times 100\%\ \text{OB} \tag{3.8.2}$$

$$W_{DS,WP} = 100 - W_{M,WP} \qquad W_{M,WP} = \text{moisture content of wet pulp} \tag{3.8.3}$$

The pressed pulp yield (M_{WP}, the mass of pressed pulp produced per 100 kg of beet) is calculated from the marc content of cossettes and the % DS of pressed pulp ($W_{DS,WP}$):

$$M_{PP} = \frac{W_{M,C}}{W_{DS,PP}} \times 100\%\ \text{OB} \tag{3.8.4}$$

where

$$W_{DS,DP} = 100 - W_{M,DP} \qquad W_{M,DP} = \text{Moisture content of dry pulp} \tag{3.8.5}$$

The mass of press water (M_{PW}) is therefore

$$M_{PW} = M_{WP} - M_{PP} \tag{3.8.6}$$

The dry pulp yield (M_{DP}, the mass of dry pulp produced per 100 kg of beet) is calculated from the mass of pressed pulp (M_{PP}), the %DS of pressed pulp ($W_{DS,PP}$), and the %DS of dry pulp ($W_{DS,DP}$):

$$M_{DP} = M_{PP} \times \frac{W_{DS,PP}}{W_{DS,DP}} \tag{3.8.7}$$

The mass of evaporated water during pulp drying (M_W) is therefore

$$M_W = M_{PP} - M_{DP} \tag{3.8.8}$$

EXAMPLE 1

Given:

Marc content of cossettes ($W_{M,C}$)	5.0%
Dry substance of wet pulp ($W_{DS,WP}$)	10.0%
Dry substance of pressed pulp ($W_{DS,PP}$)	25%

Calculate:

Mass of wet pulp (M_{WP})

Mass of pressed pulp (M_{PP})

Mass of press water (M_{PW})

$$M_{WP} = \frac{W_{M,C}}{W_{DS,WP}} \times 100 = \frac{5.0}{10.0} \times 100 = 50.0\%\ \text{OB}$$

$$M_{PP} = \frac{W_{M,C}}{W_{DS,PP}} \times 100 = \frac{5.0}{25.0} \times 100 = 20.0\%\ \text{OB}$$

$$M_{PW} = M_{WP} - M_{PP} = 50.0 - 20.0 = 30.0\%\ \text{OB}$$

EXAMPLE 2

Given:

Marc content of cossettes ($W_{M,C}$)	5.0%
Dry substance of pressed pulp ($W_{DS,PP}$)	25.0%
Dry substance of dry pulp ($W_{DS,DP}$)	90%

Calculate:

Mass of pressed pulp (M_{PP}) % OB

Mass of dry pulp (M_{DP}) % OB

Mass of water evaporated (M_W) % OB

Mass of water evaporated (M_W) % on pressed pulp

$$M_{PP} = \frac{W_{M,C}}{W_{DS,PP}} \times 100 = \frac{5.0}{25.0} \times 100 = 20.0\%\ \text{OB}$$

$$M_{DP} = M_{PP} \times \frac{W_{DS,PP}}{W_{DS,DP}} = 20.0 \times \frac{25.0}{90.0} = 5.6\%\ \text{OB}$$

$$M_W = M_{PP} - M_{DP} = 20.0 - 5.6 = 14.4\%\ \text{OB}$$

$$M_W = 14.4 \times \frac{100.0}{20.0} = 72\%\ \text{on pressed pulp}$$

NOTE

Another formula for the calculation of the mass of pressed pulp is based on the sugar content of cossettes (sugar on sugar, $W_{S,C}$):

$$M_{PP} = 1.66(W_{S,C} - 7.5)\%\ \text{OB} \tag{3.8.9}$$

The 7.5 value represents the total percentage of marc and nonsucrose substances in the beet (5.0 + 2.5 = 7.5%).

EXAMPLE 3

Given:

Sugar content of cossette ($W_{S,C}$) 17.0%

Calculate:

Mass of pressed pulp (M_{PP})

$$M_{PP} = 1.66(17.0 - 7.5) = 15.8\%\ \text{OB}$$

Sugar Loss in Pressed Pulp

The **sugar loss** in the pressed pulp (M_{SLPP_P}) from processing 100 kg of beet is equal to the mass of pressed pulp (M_{PP}) times its sugar content ($W_{S,PP}$):

$$100 \times M_{SL,PP} = M_{PP} \times W_{S,PP} \tag{3.8.10}$$

$$M_{SL,PP} = \frac{M_{PP} \times W_{S,PP}}{100} \tag{3.8.11}$$

Substituting the pulp yield M_{PP} from Eq. (3.8.11) into Eq. (3.8.4) results in the following equations:

$$M_{SL,PP} = \frac{M_{PP} \times W_{S,PP}}{100} = \frac{(W_{M,C}/W_{DS,PP})(100 \times W_{S,PP})}{100}$$
$$M_{SL,PP} = \frac{M_{M,C} \times W_{S,PP}}{W_{DS,PP}} \tag{3.8.12}$$

EXAMPLE 4

Given:

Marc content of cossettes ($W_{M,C}$)	5.0% OB
Sugar in pressed pulp ($W_{S,PP}$)	1.7% m/m
Dry substance of pressed pulp ($W_{DS,PP}$)	25.0% m/m

Calculate:

Sugar loss in pressed pulp ($M_{SL,PP}$)

$$M_{SL,PP} = \frac{M_{M,C} \times W_{S,PP}}{W_{DS,PP}} = \frac{5.0 \times 1.7}{25.0} = 0.34\%\ \text{OB}$$

Energy Balance of Pulp Drying

Energy (heat) must be supplied to the pulp dryer to

- Heat the water in the pulp to the evaporation temperature (100°C)
- Evaporate the water
- Heat the vapor to its final temperature (about 120°C)
- Heat the pulp to its final temperature (about 75°C)

The heat is mainly used for the second item; therefore, the amount of heat used for the first, third, and fourth items is often negligible. The heat required for the first, third, and fourth items can be calculated by the following equation:

$$Q = M_W C_P \Delta T \tag{3.8.13}$$

Q	Heat in kJ, Btu (for conversion: 1 kJ = 1 Btu) or kcal (1 kcal = 4.184 kJ)
M_F	Amount of water to be evaporated in kg or lb (for conversion: kg × 2.2 = lb)
C_P	Specific-heat capacity in kJ/kg.°C or Btu/lbs.°F (for conversion: kJ/kg.°C/4.187 = Btu/lb.°F)
ΔT	Change in temperature in °C or °F (for conversion: °C × 1.8 + 32 = °F)

The heat required for the second item can be calculated by the following equation:

$$Q = M_W . \lambda_W = M_W \times 2257 \tag{3.8.14}$$

M_W	Amount of water in kg or lb (for conversion: kg × 2.2 = lb)
λ_W	Latent heat of evaporation in kJ/kg or Btu/lb (for conversion: (kJ/kg)/2.3 = Btu/lb)

EXAMPLE 5

Given:

Beet capacity	200 kg/h
Pressed pulp produced (M_{PP})	100 kg/h
Dry substance of pressed pulp ($W_{DS,PP}$)	25%
Dry substance of dry pulp ($W_{DS,DP}$)	90%
Temperature of pressed pulp (T_{PP})	50°C
Temperature of dry pulp (T_{DP})	75°C
Exhaust gas temperature	120°C
Latent heat of water (λ_W) at 100°C	2257 kJ/kg

Calculate:

Amount of heat required for drying pressed pulp

In Example 2, the amount of water evaporated from 100 kg pressed pulp with 25% *DS* to dry pulp with 90% *DS* was found to be 72% on pressed pulp. Therefore, the amount of solids in pressed pulp is

$$M_P = 100 - M_W = 100 - 72 = 28 \text{ kg}$$

The heat required for heating water from 50 to 100°C is

$$Q = M_W.C_P.\Delta T = 72 \times 4.18(100 - 50) = 15048\,\text{kJ}$$

C_P Specific heat of water at 50°C is 4.18

To evaporate water,

$$Q = M_W.\lambda_W = 72 \times 2257 = 162504\,\text{kJ}$$

To heat the vapor to its final temperature (from 100 to 120°C),

$$Q = M_V.C_{PV}.\Delta T = 72 \times 2.03(120 - 100) = 2923\,\text{kJ}$$

C_{PV} Specific heat of vapor at 100°C is 2.03 kJ/kg. °C

To heat the pulp from 55 to 75°C,

$$Q = M_P.C_{PP}.\Delta T = 28 \times 2.10(75 - 55) = 1176\,\text{kJ}$$

where

C_{PP} Specific heat of pulp is 2.10 kJ/kg.°C

The total heat required is

$$15048 + 162504 + 2923 + 1176 = 181651\,\text{kJ}$$

This is equal to 2523 kJ per kg of evaporated water.

OPERATING PROBLEMS

This section describes some **problems** that can occur in pulp-pressing and pulp-drying operations.

Low Pulp-Pressing Efficiency

Newly developed high-pressure pulp presses produce pressed pulp with a reasonably dry-substance content (about 30% *DS*). However, the following are causes of low pressability:

- **Wrong pulp particle size:** Cossette size, in particular, its thickness, affects the pulp-pressing operation. Thin cossettes produce thin pulp particles, which are harder to press than thick pulp particles. On average, cossettes with 4 mm thickness produce pulp particles with optimum pressability.
- **Wrong diffuser temperature:** Operating the diffuser at a high temperature (above 73°C) decreases pulp rigidity and pressability.
- **Wrong pulp temperature:** The optimum temperature of wet pulp during pressing is about 30°C. A lower temperature decreases pulp pressability.
- **High pH:** The optimum pH for diffusion operations is about 6.2. Running the diffuser at a high **pH** (above 6.5) causes the pulp to soften, lowering its pressability.

Low Pulp-Drying Efficiency

The usual causes of low pulp-drying efficiency follow:

- **Wrong pulp particle size:** Thick pulp particles lose their moisture much more slowly than thin particles.
- **Low pulp dryer capacity**
- **Low temperature of combustion air**

CHAPTER 3

SECTION 9

MILK-OF-LIME AND CARBONATION-GAS PRODUCTION

SUBSECTIONS

- Importance of Milk-of-Lime and Carbonation Gas
- Limestone and Fuel
- Types of Limekilns
- Operation and Control of the Limekiln
- Recalcining Carbonation-Lime Residue
- Milk-of-Lime Production
- Material Balance
- Operating Problems

ABOUT THIS SECTION **This section** discusses **Station 9** (Milk-of-Lime and Carbonation-Gas Production) of sugarbeet processing. Station 9 (see Figure 3.9.1) prepares two important chemicals used in Station 10 (Juice Purification): **milk-of-lime** (MOL) and **carbonation gas** (CO_2 gas). Station 9 consists of two sections: **calcining** (the decomposition of limestone in the limekiln to produce quicklime) and **slaking** (the mixing of quicklime with water in the slaker to produce milk-of-lime). Raw materials for the limekiln are limestone ($CaCO_3$) and coke (C). Beet-sugar factories use considerable amount of $CaCO_3$ (2 to 6% on beet) each day. This equates to 1 to 3% CaO on beet (OB). Coke or natural gas is used as the kiln's fuel.

In this section, you learn the principles of limekiln and slaker operations to produce quality **milk-of-lime** (MOL or line) and **carbonation gas** (simply gas).

Beet-Sugar Handbook, by Mosen Asadi
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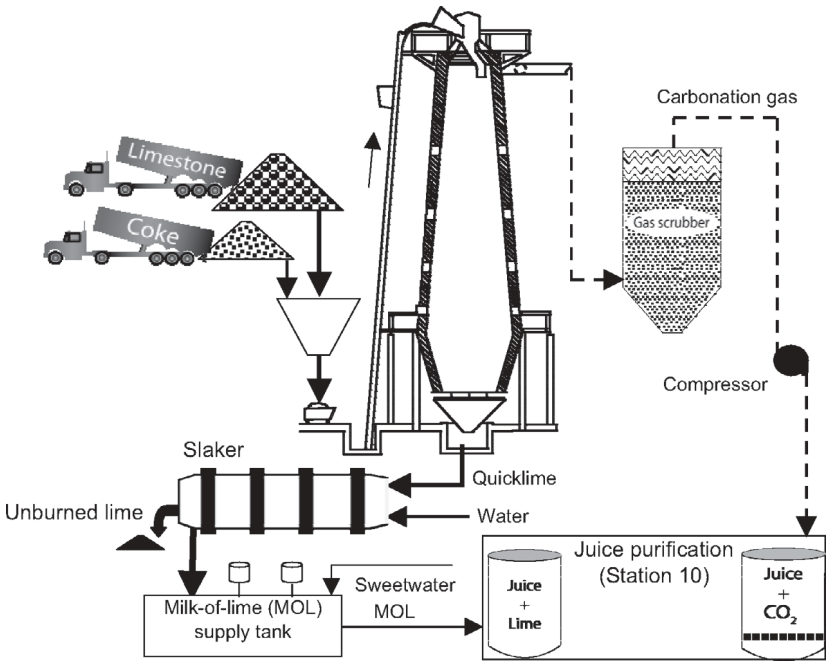


Figure 3.9.1 Flow diagram of milk-of-lime and carbonation-gas production (Station 9)

The milk-of-lime (MOL, **carbonation lime** or simply lime) and **carbonation-gas** (simply gas) station consists of two steps:

- **Calcining:** The decomposition of limestone ($CaCO_3$) to quicklime (CaO) and carbon dioxide gas (CO_2) in the limekiln at about 1 100°C by using the heat energy of the limekiln's fuel,

$$100\,\text{kg}\ CaCO_3 + \text{Heat} \rightarrow CaO + CO_2$$

- **Slaking:** The mixing of quicklime with water or a diluted juice (often sweet water from filters) in the slaker to produce lime, a suspension of $Ca(OH)_2$ in water,

$$CaO + H_2O \rightarrow Ca(OH)_2$$

The valid question here is, why can we not use calcium carbonate ($CaCO_3$) in the juice purification (Station 10) but instead must convert it to quicklime (CaO) in the limekiln? The answer is simple: $CaCO_3$ is insoluble in water and CaO is soluble. Another question is, why can we not use dry CaO in the juice-purification process but instead must mix it with water to produce liquid lime (MOL)? Liquid lime is used because it is more effective and easier to use. A third question might be, why is the CO_2 gas produced in the limekiln called **carbonation gas**? The gas produced in the limekiln is not pure CO_2 and consists of about 33% CO_2, 63% N_2, and 3% O_2. However, some sugar technologists call the gas CO_2 because CO_2 is the only component of the gas that actively participates in chemical reactions. In this book, the term **carbonation gas** (CG or simply gas) is used.

In the limekiln operation, limestone and coke are weighed automatically, mixed together into buckets in a limestone-to-coke ratio of about 10 to 1, and transported by a bucket elevator to the top of the kiln (see Figure 3.9.2). Then, the feed is discharged into the kiln through a hopper in an equal distribution.

The mixing ratio and the adjustment of air draft to the kiln are important factors in optimizing the kiln's operation. The feed moves slowly down the kiln through three zones:

- Preheating
- Calcining
- Cooling

Each zone occupies about one-third of the kiln's height. The temperature in the middle of the calcining (burning) zone is kept at about 1100°C (the hottest of the three zones) to completely convert the $CaCO_3$ to CaO and CO_2. After discharge from the bottom of the kiln, the quicklime (CaO) is transported by a belt conveyor to the **lime slaker**. In the slaker, the CaO is



Figure 3.9.2 Limekiln (Courtesy, Minn-Dak Farmers Cooperative Sugar Company)

mixed with water to produce lime. The lime is kept in a supply tank for about 30 minutes to complete the reaction between lime and water. At this point, the lime is ready to be used in the juice-purification station (Station 10).

Carbonation gas is collected at the top of the kiln and piped to the **gas scrubber** (dust collector) where lime particles are removed and the gas is cooled. The clean gas is directed to the gas compressor to increase its pressure to about 1.7 atm (170 kPa or 24.7 psi). At this point, the carbonation gas is ready for use.

Decomposition of limestone in the kiln is an **endothermic reaction**. It needs 181.2 kilojoule (kJ), equal to 43.3 kilocalories (kcal) of heat energy to decompose 1 kg of $CaCO_3$. In other words, to produce 1 kg CaO, 323.6 kJ heat energy is required (181.2 × 100/56 = 323.6). The molecular mass of CaO and $CaCO_3$ is 56 and 100, respectively:

$$100\,\text{kg}\ CaCO_3 + 181.2\,\text{kJ}\ (43.3\,\text{kcal})\ \text{Heat} \rightarrow 56\,\text{kg}\ CaO + 44\,\text{kg}\ CO_2$$

Fuel combustion supplies the heat energy required during the reaction. **Combustion** is the chemical reaction of fuel with oxygen. Oxygen for combustion is provided by air, which must be mixed with fuel in the most efficient way. The air we breathe is a mixture of nitrogen (N), oxygen (O), water (the amount depends on the weather's moisture), and a small amount of carbon dioxide (CO_2). A typical cubic volume of **dry air** has the following composition, which is used in technical calculations:

Oxygen (O)	21% by volume
Nitrogen (N)	78% by volume
Carbon dioxide (CO_2)	1% by volume

Coke, anthracite coal, oil, or natural gas can be used in the limekiln as **fuel**. Coke and natural gas are the most frequently used fuels. Carbon (C) is the main component of fuels. It reacts with oxygen (the reaction is called combustion) to produce heat:

$$C + O_2 \rightarrow CO_2 + \text{Heat}$$

Combustion is a highly **exothermic reaction**. The energy released during the reaction depends on the type of fuel. For example, the heat value (*HV*, heat of combustion) of ethane (the main component of natural gas) is about 52000 kJ/kg (22 318 Btu/lb). This means that when 1 kg of ethane burns, 52000 kJ of heat are released.

NOTE

Although calcium hydroxide [$Ca(OH)_2$] is used during juice purification, the term lime refers to calcium oxide (CaO) in sugar terms and calculations.

IMPORTANCE OF MILK-OF-LIME AND CARBONATION GAS

The **juice-purification process** (the subject of the next station) is known to sugar technologists as the **heart of the sugar factory**. Because milk-of-lime (MOL) and carbonation gas are the main chemicals used in this station, it is important to optimally manage the limekiln operation to produce sufficient quantities of high-quality lime and gas. The quality of the lime and gas largely determines the performance of a beet-sugar factory. In the operation of a limekiln, a

supply of suitable limestone and coke is important to producing high-quality quicklime and carbonation gas. High-quality quicklime has the following properties:

- Has a high degree of reactivity with water in the slaking process (slakes well)
- Contains a high amount of CaO (more than 90%)
- Contains a low quantity of silica (SiO_2)
- Is not undercalcined or overcalcined
- Is free of fuel

Carbonation gas used in the purification station should have the following specifications:

CO_2	30–35%
N_2	65–68%
O_2	2–4%
SO_2	Small amount

Density 1.5 kg/m^3

The density of gas is calculated from the densities of the CO_2 and N_2 gases. At atmospheric pressure, CO_2 and N_2 gases have densities of 1.95 kg/m^3 and 1.25 kg/m^3, respectively. Because carbonation gas consists of about 35% CO_2 and 65% N_2, its density is estimated as 1.5 kg/m^3.

The **combustion air requirement** (CAR) is the kg (or lb) of air needed for combustion of each kg (or lb) of fuel. The CAR calculation is based on fuel analyses. In practice, some **excess** (surplus) **oxygen** above the theoretical amount is used to achieve complete combustion of the fuel. The presence of oxygen in the gas is an indication of excess oxygen above the theoretical amount needed during combustion. Without enough air, the oxygen required for combustion is not available. Oxygen shortfall results in the formation of **carbon monoxide** (CO), which is *hazardous*. For efficient limekiln operation, 10 to 20% excess air over the theoretical amount is needed (*the better the air and fuel are mixed, the less excess air is needed*.) (Note: The optimum mixing of fuel and air is important in the combustion process.)

Gas leaves the kiln at about 300 to 400°C and contains some lime particles, so it has to be filtered and cooled to about 30 to 40°C in a **gas scrubber** (dust collector). The gas enters the scrubber through nozzles, which change the gas into fine bubbles. Rashing rings on the upper part of the scrubber separate the lime particles and dust from the bubbles. The water containing impurities is gradually replaced by fresh water.

LIMESTONE AND FUEL

Limestone (lime rock) exists in large quantities throughout the world in the form of stone composed of skeletons of marine animals. The chemical and physical properties of limestone depend on its origin.

Limestone should meet certain specifications with regard to its composition, size, and size distribution (uniformity) to be calcined well in the kiln. Limestone mainly consists of calcium carbonate ($CaCO_3$) and some impurities. High-quality limestone refers to its high $CaCO_3$ content and low impurities. Limestone containing $CaCO_3$ lower than 94% is not suitable for use in the limekiln. High silica (SiO_2) content (more than 2%) creates **clinker** (hard and cement-type material) in the kiln, which can block the kiln. In addition, it prevents

complete slaking or later leaves deposits in the evaporators tubes in the form of calcium silicate ($CaSiO_3$). High sulfate is not desired as well because it causes scaling in the evaporators. Limestone with high alkali (Na_2O and K_2O) content damages the refractory brick lining of the kiln. The following table indicates the typical composition of limestone suitable for use in the limekiln:

Typical Limestone Composition

$CaCO_3$	95.0% min
$MgCO_3$	2.0% max
SiO_2	2.0% max
Al_2O_3	0.4% max
$Na_2O + K_2O$	0.2% max
SO_4	0.2% max
Bulk density	1500 kg/m^3 (94 lb/ft^3)

Uniformed limestone particles with 60 to 160 mm (2.4 to 6.4 in.) across are desirable. This size of limestone weighs about 2 kg per piece. Large pieces of limestone require a longer burning time, and too-small pieces increase resistance to the flow of CG in the kiln. Limestone should be clean of clay and mud because these materials block the complete exit of the gas from the kiln.

Beet-sugar factories usually use 5% OB (on beet) limestone. For example, a factory with 8000 t capacity uses about 400 t of limestone a day (8000 × 5/100 = 400) and produces 224 t of CaO a day (400 × 56/100 = 224), or 33600 t CaO in 150 days of sugarbeet processing.

NOTE

Lime (quicklime) is used in many different industries. In the United States, about 22 million tons of lime and 5 million tons of CO_2 are produced annually.

Sugar factories mostly use **coke** and **natural gas** as limekiln fuel because these are the lowest cost fuels in term of energy content and the most suitable for limekiln operation. Some sugar factories also use **oil**.

Coke-fired kilns usually use 40/60 or 60/80 mm grade coke. In general, uniform coke pieces of 40 to 60 mm (1.6 to 2.4 in.) are suitable for use. Coke with high sulfur (S) content is not desirable because of carbonation-gas odor and other environmental difficulties. The following table indicates the composition of a suitable coke:

Suitable Coke Composition

C	80.0% min
Ash	12.0% max
O	5.0% max
N	1.0% max
S	0.5% max
H_2O	6.0% max
Bulk density	400 kg/m^3 (25 lb/ft^3)

To make a better decision regarding which fuel type is the most economical, you need to know the heat value of the fuel. The **heat value** (*HV*, also called **heat of combustion**) of a fuel determines how much **heat** (heat energy) can be produced by a unit quantity of the fuel. The following equation is used to calculate the heat value of coke based on its ash (*A*) and water (*W*) content:

$$HV = 4.184[79.24(100 - A - W) - 6W] \tag{3.9.1}$$

EXAMPLE 1

Given:

A coke sample contains:

Carbon	80.5%
Ash	12.1%
Water	2.6%

Calculate:

HV of the sample,

$$HV = 4.184[79.24(100 - 12.1 - 2.6) - 6 \times 2.6] = 28215\,\text{kJ/kg}\ (12109\,\text{Btu/lb})$$

$$1\,\text{Btu/lb} = 2.33\,\text{kJ/kg}$$

Natural gas usually contains methane (CH_4), ethane (C_2H_6), propane (C_3H_8), and a small amount of hydrogen, nitrogen, and carbon monoxide. The heat value (*HV*) of natural gas is determined by calculations based on a chemical analyses of the fuel:

$$HV = 33878\text{C} + 144460\left(\text{H} - \frac{\text{O}}{8}\right) + 9436\text{S}\ \text{kJ/kg} \tag{3.9.2}$$

$$HV = 14540\text{C} + 62000\left(\text{H} - \frac{\text{O}}{8}\right) + 4050\text{S}\ \text{Btu/lb} \tag{3.9.3}$$

C	% carbon
H	% hydrogen
O	% oxygen
S	% sulfur

NOTES

- **In the past, sugar factories purchased sufficient limestone and coke during the intercampaign to last the entire beet campaign. Today, to save on storage space, the factory receives its supply gradually**
- **Limestone and coke are stored near the limekiln to make their transportation to the kiln easier**

TYPES OF LIMEKILNS

Two types of limekilns are mainly used in the sugar industry:

- Solid-fired kilns
- Liquid-fired kilns

Solid-Fired Kiln

Several types of **solid-fired kilns** are available, but the Belgian-type kilns (known as vertical kilns) are the most popular in the beet-sugar industry. These kilns are also known as **mixed-feed shaft kilns** because limestone and coke are mixed together before they are fed to the kiln. These kilns can use coke or anthracite coal as a fuel. A Belgian kiln (see Figure 3.9.3) is a tall round shaft with a height-to-diameter ratio of about 5. Depending on the kiln's capacity, the shaft's height varies from 18 to 22 m (59 to 72 ft) and the inside diameter from 3 to 4 m (10 to 13 ft). A typical limekiln with 350 t capacity has an overall height of 55 m (180 ft) with an outside shaft diameter of 5.5 m (18 ft). To withstand the operating temperature, the inside of the kiln is lined with two layers of refractory bricks.

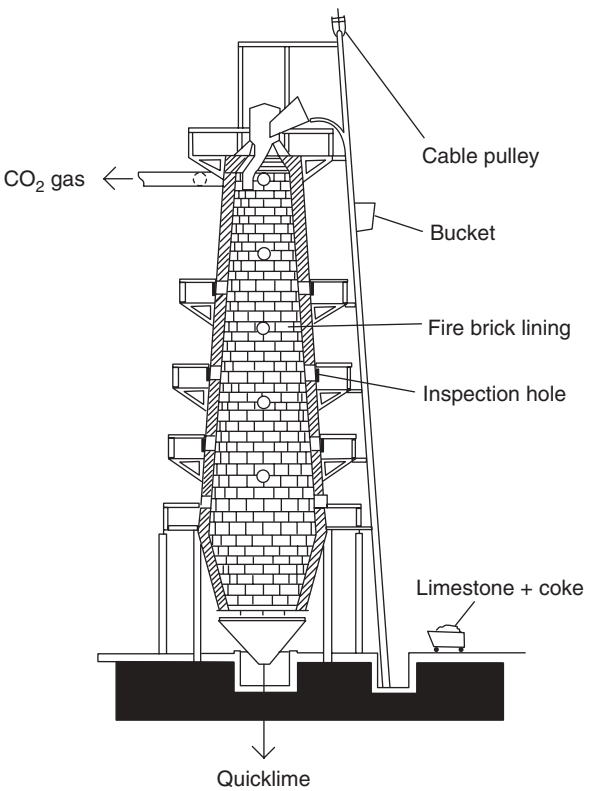


Figure 3.9.3 Coke-fired limekiln

The mixing ratio of limestone and coke is about 10 to 1 (the weight of coke is 8 to 10% of limestone). Usually, the limestone and coke are weighed by integrated scales to ensure the right weights. The weighted materials are dropped on a belt that ends in a hopper. Instead of a belt, some factories use a vibrating conveyor to mix the stone and coke more uniformly, which is critical to good kiln operation. The hopper holds about a day's worth of feed needed by the kiln. The hopper feeds the discharging buckets, which are transported by an elevator to the top of the kiln. Here, the kiln door opens, and the charge is discharged by a distributor into the kiln in a uniform fashion.

The discharging distributors are rotating or rotating-vibratory types. In the rotating distributor, the feed is first discharged to a small bin (about 1.5 times larger than a discharging bucket), which transfers the charge to a rotating chute with several arms. The arms are different in length in order to uniformly discharge a charge over a cross section of the kiln.

In the rotating-vibratory type, the chute vibrates and rotates as it releases the charge into the kiln. The feed moves down the kiln through three zones:

- Preheating
- Calcining
- Cooling

Each zone occupies about one-third of the kiln's height. The exact height of the zones in relation to the total height of the kiln cannot be determined because the calcining (burning) zone moves by changing the following variations:

- Volume of the air entered into the kiln
- Quality and size of the limestone
- Heat value of fuel

The amount of the air entering the kiln is important to the kiln's operation. For example, if the amount of air is not enough for fuel combustion, the calcining zone will move lower than its normal level, and the lime will become undercalcined.

In the kiln, the batches of the feed (limestone and coke) keep moving down through the preheating zone, which absorbs heat from the gases moving up from the calcining zone. When the feed reaches the calcining zone, it starts to burn in the high heat. Here, the limestone decomposes into CaO and CO_2 gas. When the feed reaches the cooling zone, it is cooled gradually by incoming air.

At the bottom of the kiln, a controlled amount of the calcined lime (quicklime) is discharged continuously from the kiln by a movable grate to a hopper and transported to the slaker by a conveyer belt. The discharge of burned lime makes space for new batches of the feed. In addition, during decomposition about 44% of the limestone mass is converted into CO_2 gas, which frees up more space for feed. The discharged lime is fed to the hopper and from there to the conveyer, which directs the quicklime to the slaker.

Most limekilns in sugar factories depend on **mechanical draft** (induced draft), in which air is forced from the bottom of the kiln by a fan. The **draft** is the difference in pressure between two points that creates the flow of air from higher pressure to lower pressure.

The gas is continuously withdrawn at the top of the kiln by a **compressor**. The compressor has a variable speed to suit changing conditions. The compressor also helps adjust the

amount of air entering the kiln. After leaving the kiln, the gas goes through the **dust collectors** (scrubbers) for dust and lime-particle filtration and then is supplied to the carbonation process.

Liquid-Fired Limekiln

Because of their high capital cost, the use of **liquid-fired kilns** is not economical for sugar factories. Conversion of coke-fired kilns to oil or gas has been tried. It seems that the conversion-fuel system is economically the more attractive choice for the beet-sugar industry. Use of these kilns is particularly attractive when gas or oil prices are cheaper per heat unit than coke and cover the expense of conversion. In general, the liquid-fired kiln consists of a shaft similar to a solid-fired kiln. The fuel is introduced to the bottom of the kiln through **gasification chambers**. Air for combustion is introduced from below the chambers. In oil firing, additional equipment is required to convert the fuel oil to gas before it enters the chamber.

Following are the advantages of the liquid-fired kiln over the solid-fired kiln:

- Lower fuel cost
- Better fuel distribution
- Less sensitivity to limestone quality (composition and size)
- Elimination of ash (in case of gas use) or its reduction (in case of oil use)

The disadvantages are just as many:

- High capital cost
- Difficulties in controlling the limestone-to-fuel ratio
- Lower heat efficiency (about 60% in oil and 55% in gas compared with 70% in solid-fired kilns)
- Lower CO_2 production since liquid-fired kilns produce gas that contains less CO_2 (about 30% CO_2 by volume compared with about 35% in solid-fired kilns)

The difficulties in controlling the limestone-to-fuel ratio can cause underburning or overburning of the lime. However, this problem is overcome in some gas-fired kilns by the installation of a hopper scale under the kiln, which controls the discharge from the kiln.

OPERATION AND CONTROL OF THE LIMEKILN

The **limekiln** is usually fired three to four days before the start of the beet campaign (*the normal limestone burning time is 25 to 30 hours*). If the kiln was emptied completely at the end of the previous campaign, it is fed first with limestone up to the cooling zone. Solid-fired kilns are shut down at the end of each campaign, keeping the level of feed in the kiln to the cooling zone.

To fire the kiln, a layer of wood and coal (about 50 kg) is introduced into the kiln from the top and ignited with a lighter soaked in kerosene. Then, almost a ton of coke is added, following a batch of feed consisting of 70% coke and 30% limestone. The next few batches contain less coke until the kiln reaches a regular feed of about 10 parts limestone and 1 part coke.

The following are a few important recommendations for operating a limekiln:

- **Limestone-fuel ratio:** In solid-fired kilns, the quantity of the coke should be 8 to 10% per mass of limestone. When this ratio is below 8%, the kiln temperature is lower than normal,

causing problems such as underburning the lime and lowering CO_2 gas production. When the quantity of the coke is too high, the kiln temperature exceeds the limit, causing problems such as overburning of lime, the formation of clinkers (at very high temperatures, fusible silicates form clinkers that may stick together and to kiln bricks, causing blockages), and damage to the refractory bricks of the kiln.

- **Kiln temperature:** Complete decomposition of limestone cannot take place at temperatures lower than 900°C (exactly 882°C at atmospheric pressure). Because the pressure in a kiln is higher than the atmospheric pressure, the temperature in the middle of calcining zone of the kiln should be kept at about 1100°C to speed the decomposition of the limestone. At high temperatures (above 1300°C), silicate present in the limestone reacts with calcium and aluminum to produce calcium and aluminum silicates, which prevent the complete **slaking process** (reaction of CaO with water to produce MOL). Therefore, the optimum temperature for limekiln operation is 1050 to 1150°C. (Note: The pressure of the CO_2 gas produced depends on the kiln temperature (*the higher the temperature, the higher the pressure of the gas.*)
- **Steady CO_2 removal:** Gas should be removed continuously as it is produced so that it does not recombine with the finished lime (CaO).
- **Steady rate of input and output:** No more lime should be taken than corresponds to the limestone fed to the kiln. This is roughly 50% by mass. The rates of feed input (limestone and coke) and output (quicklime and carbonation gas) are important in kiln operation because the height of the calcining zone should be kept constant as much as possible in relation to the total height of the kiln. If the quantity of the input is decreased, the fixed air will be excessive and the calcining-zone level will rise. On the other hand, if the quantity of the output is increased, there will not be enough air and the calcining-zone level will drop.
- **Venting surplus gas:** There might be cases where CO_2 gas is in excess of what the purification station needs. In the case of gas surplus, it is best not to disturb the rate of gas withdrawal. The excess gas can be vented to the atmosphere, because it is environmentally friendly.

The temperatures of the different zones, particularly the calcining zone, must be controlled in the kiln during operation. The temperatures are usually maintained by thermocouple-type thermometers installed in different places in the kiln. For example, by installing three or four thermometers in different parts of the calcining zone, you can control the temperature and maintain the correct amount of quicklime discharge from the kiln.

The fill level of the kiln can be controlled by a gamma-ray level probe, which is installed on the charging area of the kiln.

RECALCINING CARBONATION-LIME RESIDUE

Recalcining is the process of reusing **carbonation-lime** residue (also known as **limecake**, the by-product of the purification process) again in the purification station. In the next section (Station 10), you will learn that during purification, the diffusion juice is limed (meaning milk of lime [$Ca(OH)_2$] is added). You will also learn that the high amount of lime used during the purification process (1.0 to 2.5% on beet) converts again to calcium carbonate ($CaCO_3$) when the limed juice is carbonated with carbonation gas:

$$CaO + CO_2 \rightarrow CaCO_3\downarrow$$

The precipitated calcium carbonate is separated from the juice in the clarifier, dewatered (to 30 to 50% moisture content) in the cake filters (filter presses or rotary-vacuum filters), and piled in the lime pond as **carbonation-lime residue**(CLR, limecake or used calcium carbonate). Calcium carbonate produced as such is not the same as the original (fresh) calcium carbonate because of impurities (nonsugars) absorbed during the purification.

In some areas, the cost of limestone is so high that the factory invests in equipment to recalcine the carbonation-lime residue to be reused in the purification station. The main equipment of the recalcining process is the kiln (mostly, rotary limekiln) in which the lime is burned at about 1100°C to produce quicklime (CaO) and then cooled in a heat exchanger. The lime from the re-burned lime residue has more impurities than lime generated by burning limestone, so some virgin limestone is often burned with lime residue to increase its lime content. The recalcined quicklime is then slacked to produce milk of lime the same way as the quicklime produced from fresh limestone. The milk-of-lime production is explained next.

MILK-OF-LIME PRODUCTION

Quicklime (CaO) produced in the limekiln is not completely suitable for use in the purification station. Therefore, it is slaked (mixing quicklime with water) in warm condensate or sweet water from purification filters to produce MOL (milk-of-lime), which is a suspension (slurry) of $Ca(OH)_2$ in water. The process is called **slaking**, and the equipment used for this process is called a **slaker**. The reaction occurring during the slaking process can be formulized as follows:

$$CaO + H_2O \rightarrow Ca(OH)_2 + 1\ 092\,kJ\ (261\,kcal)$$

The reaction is highly **exothermic**, releasing 1 092 kJ energy when 1 kg of CaO reacts with warm water.

The following points should be considered in MOL preparation:

- The reactants (CaO and water) should be mixed in such a ratio as to produce MOL with about 42% dry substance (about 23 baume [BE]).
- The temperature of the water used for slaking should be kept in the range of 28 to 30°C to achieve MOL with a temperature of about 80°C, since the reaction of CaO and water releases some heat.
- The sugar content of sweet water used for slaking must not contain more than 5% dry substance (*DS*).
- During slaking, the suspension should be stirred constantly.
- After slaking, a holding time of about 30 minutes is required for the complete reaction of quicklime and water.

The slaker used in most factories consists of a horizontal drum, rotating slowly (5 to 6 RPM) on supporting rollers. MOL and water enter the feed-end, where they are mixed with paddles mounted on the inside of the drum. The unreacted lumps also move to the discharge-end, where they are discharged onto a platform. The MOL exits from the same end, and then it flows through a screen into a sand catcher. From the sand catcher, the MOL is pumped into a dilution tank to be diluted with **sweetwater** (supplied by first-carb filters) to about 42% *DS* (23 Be), see Table A.1 in the Appendix]. Such an MOL contains about 23% CaO and its density is about 1200 kg/m^3 (Kadlec et al. 1984). The product should stay in the tank for about 30

minutes to complete the reaction between lime and water. At this point, the MOL contains more than 85% **reaction activity** and is ready to be used in the purification station.

MATERIAL BALANCE

The amount of **limestone** and coke required for the limekiln's operation, fuel's heat value, and the heat efficiency have to be controlled. The following table of factors is helpful for limekiln calculations and material balance:

	Density (kg/m^3)	Molecular Mass
O	1.43	16
C		12
CO_2	1.95	44
N	1.25	23
CO	1.25	28
Air (O and N)	1.29	
Carbonation gas	1.5	
$CaCO_3$ (medium-size powder)	2700	100
CaO (medium-size powder)	2600	56
Milk of lime	1200	74
Limestone ($CaCO_3$, bulk)	1440 (90 lb/ft^3)	
Quicklime (CaO, bulk)	900 (56 lb/ft^3)	
Coke (C, bulk)	400 (25 lb/ft^3)	

Mass of CaO = Mass of $CaCO_3 \times 0.56$
Mass of CO_2 from $CaCO_3$ = Mass of $CaCO_3 \times 0.44$
Mass of CO_2 from coke = Mass of $C \times 44/12$
Volume of CO_2 from 1 kg $CaCO_3$ = 0.44/1.97

As stated, 1 666 kJ of heat is required to decompose 1 kg of $CaCO_3$. Theoretically, the quantity of coke, which has a **heat value** (*HV*) of 29 000 kJ/kg, needed for 100 kg of $CaCO_3$ is

$$HV = 100\,\text{kg} \times \frac{1666\,\text{kJ/kg}}{29000\,\text{kJ/kg}} = 5.7\,\text{kg (or \% coke on } CaCO_3\text{)}$$

But, in practice, we need 8 to 10 kg of coke because some heat is lost (mainly by conduction) during operations. For example, if we use 8 kg coke per 100 kg of limestone, the **heat efficiency** (*HE*) of the kiln is

$$HE = \frac{5.7 \times 100}{8} = 71.3\%$$

The remaining 29.7% is the heat loss, which mainly remains in carbonation gas, and the rest is lost by conduction. (Note: The total amount of CO_2 gas produced in the limekiln is equal to both reactions: *limestone decomposition* and *fuel combustion*.) The amount of CO_2 produced from 100 kg of $CaCO_3$ is 44 kg:

$$CaCO_3 + \text{Heat} \rightarrow CaO + CO_2$$

In addition, during combustion, coke also produces CO_2 gas:

$$C + O_2 \rightarrow CO_2$$

If, for example, the amount of coke used is 8% on limestone (8 kg of coke) and it contains 80% carbon, the amount of CO_2 produced is

$$CO_2 = 8 \times \frac{80}{100} \times \frac{44}{12} = 23.5\,\text{kg}$$

Therefore, the total amount of available CO_2 is 44 + 23.5 = 67.5 kg.

We also know that in the purification station, we need 44 kg of CO_2 to convert the CaO back to $CaCO_3$:

$$\underset{56}{CaO} + \underset{44}{CO_2} \rightarrow \underset{100}{CaCO_3}$$

As seen, there is excess gas in the amount of 23.5 kg (67.5 – 44 = 23.5 kg). In practice, the purification needs extra gas because the **coefficient of gas utilization** (K_{GU}) in the carbonation process (first and second carb) is lower than 100%. Using the preceding data, the K_{GU} can be calculated as

$$K_{GU} = \frac{\text{Theoretical gas need}}{\text{Available gas}} \times 100 = \frac{44}{67.5} \times 100 = 65.2\%$$

As stated, carbonation gas contains about 35% CO_2, 65% nitrogen, and 2% oxygen. The amount of oxygen required to burn coke can be calculated from the coke use (8 to 10% on limestone). The following example shows how the amount of oxygen is calculated.

EXAMPLE 2

Given:

Coke used	8.0% on limestone
Carbon (molecular mass) in coke	80.0%

Calculate:

Amount of oxygen

$$O_2 = 8.0 \times \frac{32}{12} \times \frac{80.0}{100} = 17.1\%\ \text{on limestone}$$

Note that 23% (m/m) of dry air is oxygen and 77% is nitrogen. Therefore, the amount of N_2 in carbonation gas will be

$$N_2 = 17.1 \times \frac{77}{23} = 57.2\%\ \text{on limestone}$$

As stated, at atmospheric pressure, the density of CO_2 is 1.95 kg/m^3 and that of N_2 is 1.25 kg/m^3. Therefore, the volume (V) of CO_2 and N_2 gas produced from 100 kg of limestone can be calculated as follows:

$$V_{CO_2} = \frac{67.5}{1.95} = 34.6\,m^3$$

$$V_{N_2} = \frac{57.2}{1.25} = 45.8\,m^3$$

The total carbonation gas is the sum of the preceding:

$$V_{CO_2} + V_{N_2} = 34.6 + 45.8 = 80.4\,m^3$$

Assuming that the limestone used for kiln operation is 95% pure (contains 95% $CaCO_3$) and losses are equal to 10%, the amount of limestone needed to produce 100 kg (ton or any other mass unit) of quicklime (CaO) is

$$100 \times \frac{100}{56} \times \frac{100}{95} \times \frac{100+10}{100} = 206.8\text{kg}$$

Note: The amount of limestone is a little more than double that of CaO (206.8 kg $CaCO_3$ is needed to produce 100 kg of CaO). Experience indicates that we need about 2.0 to 2.5 kg CaO for the purification station to process 100 kg of sugarbeet. Therefore, the amount of limestone needed to process 100 kg of sugarbeet is equal to $2.0 \times 206.8/100 = 4.1$ kg (or % on beet) or $2.5 \times 206.8/100 = 5.2$ kg (or % on beet).

As stated, the amount of coke required is 8 to 10% on limestone, which will be $4.1 \times 8/100 = 0.33$ or $5.2 \times 10/100 = 0.52$% on beet (% OB).

EXAMPLE 3

Given:

Capacity of a beet factory 8000 t/day

Calculate:

Limestone required when it is used at 5% OB and contains 95% $CaCO_3$
CaO produced
CaO % OB
Coke required, when coke is used at 9% on limestone
Oxygen required for combustion, when coke contains 80% C
CO_2 gas produced from limestone
CO_2 gas produced from coke
N_2 gas produced
Total carbonation gas produced

$$CaCO_3 = 8000 \times \frac{5}{100} = 400\,t$$

$$CaO = 400 \times \frac{100}{56} \times \frac{100}{95} = 752\,t$$

$$\text{Coke} = 400 \times \frac{9}{100} = 36\,\text{t}$$

$$O_2 = 36 \times \frac{32}{12} \times \frac{80}{100} = 76.8\,\text{t}$$

$$N_2 = 76.8 \times \frac{77}{23} = 257.1\,\text{t} \quad 77\% \text{ of air is } N_2$$

$$N_2 = \frac{257.1}{1.25} \times 1000 = 205680\,\text{m}^3 \quad \text{Density of } N_2 = 1.25\,\text{kg/m}^3$$

$$CO_2 = 400 \times \frac{44}{100} = 176.0\,\text{t from limestone}$$

$$CO_2 = 36 \times \frac{80}{100} \times \frac{44\,(\text{kg/kmole } CO_2)}{12\,(\text{kg/kmole C})} = 105.6\,\text{t from coke}$$

$$\text{Total } CO_2 = 176.0 + 105.6 = 281.6\,\text{t}$$

$$\text{Total } CO_2 = \frac{281.6}{1.95} \times 1000 = 144410\,\text{m}^3$$

$$\text{Total carbonation gas} = CO_2 + N_2 = 144410 + 205680 = 350090\,\text{m}^3$$

OPERATING PROBLEMS

For the production of quality lime and carbonation gas, it is important to optimize the purification station. For the effective operation of a limekiln, a supply of suitable limestone and coke is needed. This section describes some **problems** that can occur in limekiln and slaker operations.

Limekiln Low Efficiency

The following are usual causes of a limekiln's low efficiency:

- **Limestone low quality:** If the amount of $MgCO_3$ in the limestone is more than 3%, the alkalinity of the first carbonation should be kept above 0.1 g CaO in 100 mL of juice. Otherwise, the high amount of released **magnesium cations** (Mg^{2+}) will cause filtration problems. You can get a rough idea of the selection of suitable limestone by analyses in the laboratory, but the limestone's ultimate suitability cannot be known until you observe the results in the factory. Limestone that produces quicklime that slakes easily and that produces gas with enough CO_2 content (30 to 35% CO_2) is the best for kiln operation. Therefore, one or two carload trials (to cover about one week of kiln operation) are usually well worth the cost before making a decision regarding limestone selection.
- **Kiln low temperature:** As stated, the temperature of the calcining zone should be kept between 1050 and 1150°C. At this temperature, the color of the mixture in the hottest part of the calcining zone is *light red* or *dark orange*. One of the main reasons for low kiln tem-

perature is an insufficient amount of coke, which should be 8 to 10% per mass of limestone. When this ratio is below 8%, the kiln temperature is lower than normal, producing under-calcined lime and low CO_2 gas.

- **Insufficient kiln capacity:** This problem is usually seen in factories that expanded to increase their capacity but did not reconstruct the limekiln to handle the required limestone.
- **Wrong size limestone:** Uniform limestone particles measuring 60 to 160 mm (2.4 to 6.4 in.) across should be used in the limekiln. This size of limestone weighs about 2 kg per piece. Large pieces require a longer burning time, and too-small pieces increase the resistance to the flow of carbonation gas.

Too Much Unburned Lime

When lime is not completely burned (too much unreacted lime), the efficiency of the kiln is reduced. (Note: Unburned lime can be used again, but it should be mixed with new limestone before it is fed to the kiln.) The usual causes of too much unburned lime are

- Insufficient oxygen
- Insufficient coke use
- Short combustion time
- Limestone pieces that are too large
- Incomplete mixing of limestone and coke

Incomplete Coke Burning

The usual causes of incomplete coke burning follow:

- **High amount of coke:** When the amount of coke in the feed mixture is high (more than 10%), part of the coke cannot be completely combusted. So the kiln produces small pieces of unburned coke.
- **Too large or too small coke pieces:** Uniform coke pieces measuring 40 to 60 mm (1.6 to 2.4 in.) are suitable for use. Large pieces of coke continue burning even when they pass the calcining zone, causing the calcining and cooling zones to move. Therefore, calcined lime discharged from the kiln is warmer than usual. (Note: The temperature of normal calcined lime discharged from the kiln can be touched without burning your hands.) Small pieces of coke burn faster than usual, causing insufficient air, an incomplete combustion process, and an increased calcining-zone temperature.

Low CO_2 Content

Carbonation gas used in the purification station should contain 30 to 35% CO_2. Gas with a low CO_2 content is due to the following:

- Unsealed gas outlet
- Unsealed gas scrubber
- Insufficient combustion air
- High temperature in the calcining zone
- Top door of the kiln stays open longer than usual while the kiln is fed

Slaker Low Efficiency

The possible causes of slaker low efficiency (slaker produces low-quality lime) are as follows:

- Low-quality quicklime
- Too much unburned lime entering the slaker
- Insufficient water or sweet water to the slaker
- Short reaction time in the slaker (optimum is 15 minutes)
- Short holding time in the lime tank (optimum is 30 minutes)
- High sugar content of solution used during the slaking process (sweet water used for slaking must not contain more than 5% dry substance and 4% sugar)
- Inappropriate temperature of the water used for slaking (this should be kept at about 30°C to achieve lime with a temperature of about 80°C because the reaction of CaO and water releases some heat)

CHAPTER 3

SECTION 10

JUICE PURIFICATION

SUBSECTIONS

- Importance of Juice Purification
- Lime Requirement
- Alkalinity and Hardness
- Solubility of CaO and CO_2
- Classical Purification
- Defeco-Carbonation Purification
- Purification Systems
- Adding Chemicals during Purification
- Purification Startup Procedure
- Purification of Damaged Beets
- Product and By-product of the Purification Station
- Microfiltration and Ultrafiltration of the Juice
- Operating Problems

ABOUT THIS SECTION **This section** explains **Station 10** (Juice Purification) of sugarbeet processing. Station 10 (see Figure 3.10.1) is the most important station of the sugar factory, known to sugar technologists as the **heart of the factory**. A sugar factory with a purification problem cannot perform well even if other stations work well. (Similarly, a sugar technologist without sufficient knowledge of the juice-purification process is not a good sugar technologist.)

The diffusion juice from the diffusion process (Station 7) contains a considerable amount of nonsugars (nonsucroses). In the juice-purification station, **milk of lime** (MOL) is added to the heated diffusion juice in a few steps to precipitate and destabilize the nonsugars. **Carbonation gas** (GC or CO_2 gas) is also added in two steps to precipitate the lime as calcium carbonate ($CaCO_3$) and to adjust the pH and alkalinity of the juice. During operation, the **precipitated calcium carbonate** (PCC) is separated from the juice in the clarifier, dewatered in the cake filters (filter presses or rotary-drum filters), and sold as **carbonation-lime** residue (CLR, one of the by-products of the factory) in soil fertilizer, as a pH adjuster, and for other uses.

Beet-Sugar Handbook, by Mosen Asadi
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The purpose of juice purification is to remove certain nonsugars, suspended particles, and **colloids** (e.g., dextran and colorants with high-molecular mass) from the diffusion juice to produce a high-purity, colloid-free, and low-color juice with minimum **hardness** (limesalts). During a typical purification process, a nonsugar elimination (*NSE*) efficiency of 20 to 30% is achieved, and the remaining nonsugars become destabilized to the point where they are harmless to the later operation and finally end up in molasses. **Thin juice** is the product of the purification station, and **carbonation-lime residue** is its by-product.

Because the number of nonsugars eliminated during juice purification is high, the reactions between them and lime are also numerous, making the chemistry of purification the most complicated in sugarbeet processing. Considering the complexity and importance of this process, sugar technologists, particularly newcomers, should pay special attention to this section.

In this section, you will learn the chemistry of the juice-purification process, guidelines for its optimum operations, and two important aspects involved with the process: **alkalinity** and **hardness** (limesalts). You will also learn how to cope with operating problems you may face while operating the juice-purification station.

In chemistry, purification is defined as improving **purity** (cleanliness or the opposite of impurity) of a compound by chemical or physical ways. In sugar technology, **juice purification** is defined as improving purity and processability of the diffusion juice by adding **milk-of-lime** (MOL or simply lime) and **carbonation gas** (simply gas) to the juice. The purification of the diffusion juice occurs in a two-step operation:

- Liming
- Carbonation

In the **liming** step, the diffusion juice is limed (i.e., lime is added) to a certain alkalinity and pH to precipitate some nonsugars. For example, sodium oxalate reacts with lime to precipitate (↓) calcium oxalate (CaC_2O_4):

$$Ca(OH)_2 + Na_2C_2O_4 \rightarrow CaC_2O_4\downarrow + 2NaOH$$

In the **carbonation** step, the limed juice is carbonated (i.e., gas is added) to a certain alkalinity and pH to convert the unreacted lime to precipitate calcium carbonate:

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3\downarrow + H_2O$$

The precipitated calcium carbonate (PCC) is removed from the juice by **settling and filtration** (the subject of Station 11) to produce clear juice. The goals of juice purification are as follows:

- Removal of all insoluble substances
- Removal of certain soluble substances (nonsugars)
- Production of a thermostable juice with minimum hardness

In the purification station, 20 to 30% of nonsugars, such as invert sugar, colloids (see the Glossary for a definition), and coloring substances, are removed. As a rule, each kg (ton or any mass

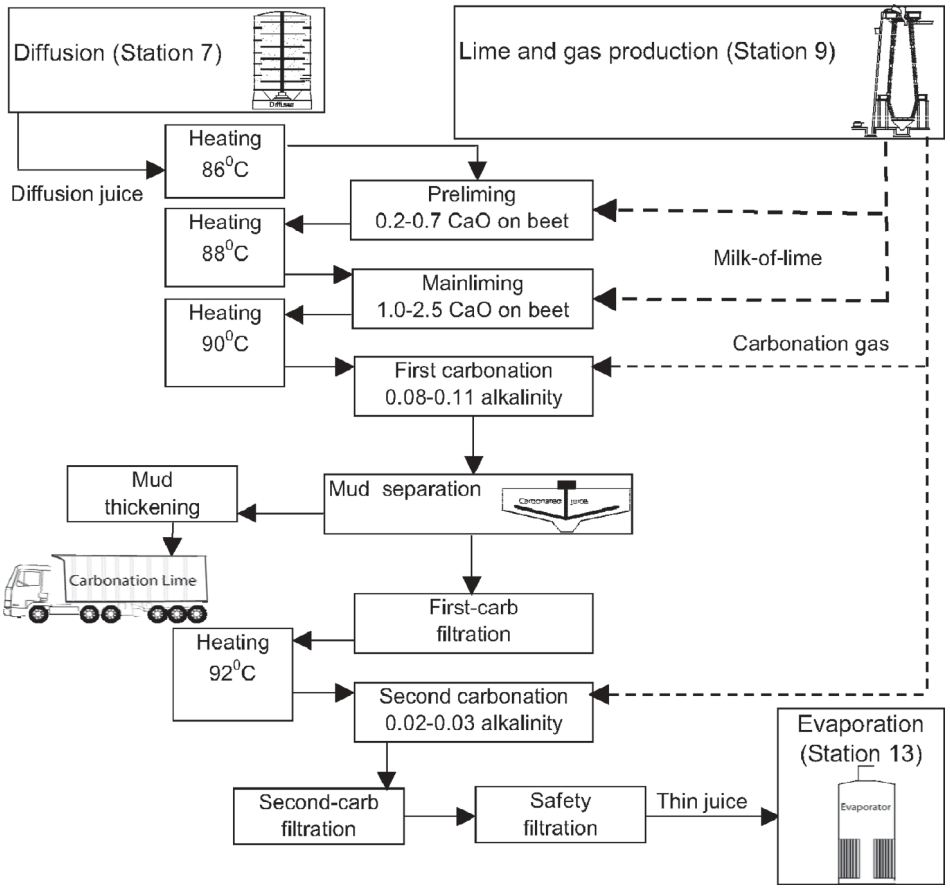


Figure 3.10.1 Flow diagram of juice purification (Station 10)

unit) of nonsugars carries 1.5 kg (ton or any mass unit) of sugar to molasses. This means that nonsugar elimination saves the factory 30 to 45% of sugar. In addition, during purification, all insoluble substances (diffusion juice contains about 2% insoluble solids) are removed, making further operations easier. The product of the purification station is a juice, known as **thin juice**, which has the following properties:

- Low color
- High purity
- Minimal hardness
- Free of insoluble solids
- Adequate settling and filterability
- Adequate thermostability (resistance against heating without alteration)

The amount of lime (see the following Notes) added to the juice during the purification process is important. Insufficient lime use is generally indicated by sedimentation and filtration

difficulties and the production of a high-color juice with high hardness content from undamaged beets. The optimization of lime usage during purification will be discussed later, but it should be obvious at this point that the lime requirement depends on the sugarbeet quality. To process healthy beet, CaO consumption of 1.0 to 2.5% on beet (OB) is the norm, which is equal to 2.0 to 5.0% limestone OB.

In addition to sufficient addition of lime and gas, the temperature, pH, and alkalinity of the juice during liming and carbonation should be carefully controlled to achieve a proper nonsugar elimination. The reaction of lime with **nonsugars** (*NS*) is complex because, during liming, different nonsugars behave differently. According to their reaction with lime, nonsugars can be divided into the following four groups:

- *NS* that react with lime to produce a precipitate
- *NS* that do not react with lime but are destabilized
- *NS* that do not react with lime but are decomposed
- *NS* that do not react with lime under any circumstances

The first three groups are **removable nonsugars** (RNS), and the fourth group contains **nonremovable nonsugars** (NRNS). Oxalates, phosphates, and sulfates present in the diffusion juice are good examples of the first group. Colloids are examples of the second group. Colloids do not precipitate with lime but destabilize (deform) to produce **colloid calcium carbonate** (CCC). The CCC is then removed from the juice by sedimentation and filtration.

Invert sugar is the best example of the third group. Invert sugar does not react with lime but is decomposed by lime to form lactic acid and other acids. The acids then precipitate with lime and are removed from the juice by filtration.

Raffinose, betaine, and amino acids are examples of the fourth group (NRNS). Nonremovable nonsugars do not react with lime under any conditions, so the NRNS stay in the juice throughout the remaining operations and end up in molasses.

Removable nonsugars (RNS) end up in the PCC and finally exit the process in the carbonation-lime residue. RNS count for about one-third of the total nonsugars in the juice (*only about one-third of nonsugars have the **right chemistry** to precipitate with lime*). At normal operation and when high-quality beets are processed, the following rates of RNS elimination are expected:

Colloids	90 to 95%
Invert sugar	80 to 90%
Pectins	65 to 75%
Organic nonsucroses	55 to 65%
Inorganic nonsucroses	30 to 35%
Nitrogenous compounds	20 to 30%

As stated, juice purification consists of the liming and carbonation processes. In classical (traditional) juice purification, the liming process takes place in two steps: **preliming** (predefecation) and **mainliming** (main defecation). Carbonation also consists of two steps: first carbonation and second carbonation (simply, first carb and second carb).

Before we get to the details of the purification process, it is helpful to outline the classical juice-purification process (for orientation, see Figure 3.10.1), which consists of the following 13 steps:

1. **Diffusion-juice heating:** The diffusion juice is heated to about 86°C.
2. **Preliming:** Lime (at a small amount of 0.2 to 0.7% OB) is added to the juice to reach an alkalinity of about 0.15% CaO and about 8.5 pH value.
3. **Prelimed-juice heating:** Prelimed juice is heated to about 88°C.
4. **Mainliming:** More lime is added to the juice (1.0 to 2.0% OB) to obtain a pH of about 12.0 to complete the reactions of the nonsucroses with the lime.
5. **Limed-juice heating:** Limed juice is heated to about 90°C.
6. **First carbonation:** Carbonation gas is added to the juice to reach a pH of about 10.8 to precipitate the excess lime and limesalts (hardness).
7. **Mud separation:** The precipitate from the first-carb juice is separated by using mud-separation equipment, such as clarifiers or filter thickeners, to produce clear juice and a thicker product, called **carbonation mud**.
8. **Mud thickening:** The mud is further thickened in **cake filters** (rotary-drum filters or filter presses) to produce limecake, also known as **carbonation-lime residue** (a by-product of the beet-sugar factory).
9. **First-carb filtration:** The clear juice from clarifiers (or thickening filters) is filtered with the first-carb filters.
10. **First-carb juice heating:** The filtered juice is heated to about 92°C.
11. **Second carbonation:** More gas is added to the juice to reach a pH of a about 9.0 to precipitate (as much as possible) the excess lime and limesalts.
12. **Second-carb filtration:** The juice is filtered by second-carb filters.
13. **Second-carb safety filtration:** The filtrate is filtered again by safety filters to prevent any fine, suspended solids entering the evaporators. (Note: Not all factories are equipped with safety filters.)

Thin juice (the product of the purification station) has a higher purity (by 2 to 5 units) than diffusion juice but a lower dry substance (by about 0.5 units). For thin juice, the typical purity value ranges from 85 to 92% and dry substance (*DS*) from 14 to 15%. Thin juice with the following properties is desired:

- pH of 8.8 to 9.0
- Low in hardness
- High in purity
- Low in color

NOTES

- **Lime in sugar terms and in calculations refers to calcium oxide (CaO), although the liming agent used during juice purification is in the form of calcium hydroxide, $Ca(OH)_2$, known as milk-of-lime (MOL). MOL contains about 23% CaO, and its density is about 1200 kg/m^3.**
- **Sugar technologists usually use the shortened form of the word carbonation, which is carb. For example, first-carb juice and second-carb filters.**
- **When we talk about alkalinity, we are referring to the alkalinity of the filtered sample. The alkalinity of a filtered-limed juice sample differs from that of an unfiltered sample. For example, if the alkalinity of a filtered-prelimed juice is 0.08% CaO on juice, that of an unfiltered sample could be as high as 0.3%.**

The pH values are based on standard room temperature (20°C). Because operating temperatures during purification are always higher than the standard temperature, the control of the purification steps should be based on either the cold sample or an estimate between hot and cold samples, experimentally determined.

IMPORTANCE OF JUICE PURIFICATION

Diffusion juice (the product of the diffusion station and feed of the purification station) has about 15% dry substance (*DS*). Its purity is low (85 to 90%), meaning that there are 10 to 15 parts of nonsugars in its 100 parts of *DS*. Therefore, when the diffusion juice is not purified, the sugar yield of the factory is lower (assuming evaporation and crystallization can proceed without difficulty). Diffusion juice has a low pH (6.0 to 6.5), is acidic (equal to about 0.04% CaO), is dark gray in color, and contains sand, silt, suspended solids, and colloids. If the diffusion juice is not **purified** and **filtered**, the following serious difficulties will occur in the next processing steps:

- Filters will be clogged immediately because of the presence of colloids
- Evaporators and heaters will be fouled immediately because of scale deposition
- Transportation of the juice will be difficult because of the presence of sands and other solids
- The crystallization process will be affected because of the high viscosity of the juice (*nonsugars increase viscosity*)

Thus far, the discussion has shown that the sugar factory cannot operate without the juice-purification station. Now you will learn about the important benefits achieved by juice purification:

- **Reduced evaporator scale formation:** Scale formation in evaporators is lowered by decreasing nonsugars and hardness.
- **Increased sugar yield:** Sugar yield is increased by increasing the purity (decreasing nonsugars) of the juice (*increasing the juice purity by 1% increases sugar yield by approximately 1.5%)*.
- **Improved juice filterability:** Juice filterability is improved by colloid removal. Colloids are the main causes of filtration and sedimentation difficulties (*colloid particles are 1 to 100 nm, whereas solution particles are smaller than 1 nm in diameter.*) Lime works as a coagulant in destabilization of the colloids to produce CCC, which is then easily separated from the juice by settling or filtration. Colloids have a high, sticky surface area, so they quickly clog the filters.
- **Increased juice thermostability:** Juice thermostability is increased by increasing the pH. Besides other benefits, a higher pH prevents **sucrose inversion** (the reaction of sucrose with water in an acidic solution to produce invert sugar) at high temperatures (*sucrose is more stable in base media than in acidic media*). In the sugar industry, the inversion of sugar is considered a sugar loss. (Not only is sucrose lost, but more nonsugars are formed.)
- **Improved juice workability:** Juice workability is improved by increasing the alkalinity and **pH** of the juice. Optimal conditions for all stations, from juice purification on up, call for alkaline (basic) media. Purified juice with a pH of about 9 meets this requirement.

- **Decreased molasses yield and sugar loss to molasses:** Molasses yield and sugar losses to molasses are decreased by increasing juice purity (*increasing juice purity by one unit results in a decrease of sugar losses in molasses by about 0.2% OB*). This leads to lower **sugar losses to molasses** (the percentage of sugar in the beet that leaves the factory in molasses). Each nonsucrose substance causes a different increase in molasses production (*the lower, the better*), and consequently, a different sucrose loss that ends up in molasses. In sugar terms, this is known as the **melassigenic effect** (see Station 18). The general statement for the melassigenic effect of nonsugars is that *each kg (ton or pound) of nonsugars carries about 1.5 kg (ton or pound) of sugar into molasses*. The damage to sugar **yield** (extraction) is also 1.5%, since sugar that does not end up in the sugar silo ends up in molasses.
- **Decreased sugar color:** The color of the juice is decreased because some of the coloring substances precipitate. Calcium carbonate has a high ratio of surface area to volume. Coloring materials are nonpolar compounds of high molecular mass, so they are adsorbed at the surface of the lime.

Although the juice-purification process has several outstanding advantages, it also has two disadvantages:

- **Creation of limesalts**: Reactions between lime and nonsugars are of two types: reactions that form precipitate and reactions that form soluble salts of Ca and Mg, known as **hardness** (limesalts). The precipitates are filtered out, but some hardness even after carbonation and filtration remains in the juice, creating difficulties in the next stations if the hardness content is high. (Hardness is one of the penalties we pay for using lime as a purifying agent. We cannot get the final traces of hardness from the purified juice unless thin-juice softening process is employed; see Chapter 6.)
- **Production of carbonation-lime residue** (limecake)**:** Lime added to the juice precipitates in the form of calcium carbonate ($CaCO_3$) when the limed juice is treated with CO_2 during the carbonation process. Carbonation-lime residue, filtered off from the first-carbonation filtration, is unwanted because, if it is not sold, it is stored in the lime pond of the factory. This can create some environmental concerns.

Mathematically, nonsugar-removal efficiency during purification is expressed by a **nonsugar-elimination** (*NSE*) formula. In general and under normal operations, the amount of *NSE* depends on beet quality. With processing healthy beet, an efficiency of 20 to 30% is usually expected, but processing damaged beet may result in a nonsugar elimination of 10 to 15%. To understand the influence of juice purification on the sugar yield (sugar extraction) of a sugar factory, study the following example.

EXAMPLE 1

Given:

Beet processed	5000 t/day
Beet sugar content	17.0%
Diffusion-juice purity (P_1)	86.5%
Thin-juice purity (P_2)	89.5%

Calculate:

- Nonsugar-elimination (*NSE*) of the purification station
- Compare the sugar-end recovery (*R*) with and without purification, assuming the factory can produce sugar without purification and the molasses purity (*PM*) in both cases is 60.0%
- The tons of extra sugar that this factory would make with purification if sugar-end losses were 0.8%

You can calculate the *NSE* by using *NSE* formula (given in the Appendix) as follows:

$$NSE = \frac{(P_2 - P_1)}{P_2(100 - P_1)} \times 10000$$

$$NSE = \frac{(89.5 - 86.5)}{89.5(100 - 86.5)} \times 10000 = 24.8\%$$

The recoveries in each case (R_1 and R_2) can be compared by using the recovery equation, given in Section 2 of Chapter 1 (knowing that the purity of granulated-refined sugar is 100%):

$$R = \frac{(P_{\text{Juice}} - P_M)}{P_{\text{Juice}}(100 - P_M)} \times 10000$$

At $P_1 = 86.5$ and $P_M = 60.0$,

$$R_1 = \frac{(86.5 - 60.0)}{86.5(100 - 60.0)} \times 10000 = 76.6\%$$

At $P_2 = 89.5$ and $P_M = 60.0$,

$$R_2 = \frac{(89.5 - 60.0)}{89.5(100 - 60.0)} \times 10000 = 82.4\%$$

The difference in recovery between the two cases is equal to

$$82.4 - 76.5 = 5.8\%$$

In a 5000 ton factory, processing sugarbeet with 17.0% sugar content, such an increase in recovery can increase the sugar production by the following amount:

$$5000 \times \frac{17.0}{100} \times \frac{5.8}{100} = 49.3\,\text{t/day}$$

(Note: For difference between recovery (*R*) and extraction (*E*), refer to Section 3 of Chapter 1.)

LIME REQUIREMENT

The **lime requirement** for adequate purification depends on the beet quality, in other words, the *amount* and *nature* of the nonsugars in the beet. When processing low-quality beet, more lime is needed because the beets have more nonsugars that must be removed. The nature of nonsugars is also important because beets from one area (or even one lot from the same area)

may require less lime, while beets from another area (or even the next lot from the same area) with the same nonsugar content may need more. The amount of lime used is usually expressed in one of the following ways:

- %CaO on juice
- %CaO on beet (OB)
- %CaO on diffusion juice nonsugars

To process healthy beets, CaO consumption of 1.5 to 2.5% OB (equal to 3.0 to 5.0% $CaCO_3$ on beet) is required. (Note: Consumption may reach 3% OB or more when processing damaged beets.) Also, note that although the amount of nonsugars of diffusion juice that can be precipitated with lime (removable nonsugars RNS) is about 0.5% OB, during purification 1.5 to 2.5% CaO on beet is used. This means that the amount of CaO is threefold to fivefold. The excess is used as a filter aid.

Sometimes the amount of lime used is expressed in %CaO **on juice**, which is almost equal to %CaO on beet. This is because the amount of diffusion juice is slightly higher than the processed sugarbeet due to **draft**, which in sugar processing is kept between 110 and 120%.

To come up with a meaningful comparison between factories, it is better to express the amount of lime on diffusion-juice nonsugars (or on nonsugars in beets) because the reactions occurring during purification are dependent on the quality of the beet (the amount and nature of the nonsugars). With today's efficient purification systems, the amount of CaO added should be kept at a moderate level of about *80% of nonsugars entering the purification station in the diffusion juice*. Low-quality beets require 120% or more. Most sugar scientists (van der Poel et al. 1998) believe that decreasing lime consumption below 70% when processing good-quality beets may result in the following unwanted results:

- Decrease in juice purity
- Increase in thin-juice color
- Increase in hardness content
- Filtration and sedimentation difficulties

Because we do not have easy access to the nonsugar content of diffusion juice, it is easier to determine the lime requirement based on the **diffusion-juice purity**, which is quite accessible from routine laboratory analyses. To come up with an approximate CaO requirement based on the purity of diffusion juice, see the next example.

EXAMPLE 2

Given:

Diffusion-juice dry substance (*DS*)	15.0%
Diffusion-juice purity (*P*)	88.0%
Diffusion draft (*D*)	115%

Calculate:

Amount of nonsucroses (*NS*) in the juice

Amount of lime (as CaO) required (% OB)

Amount of MOL ($d = 1200\,kg/m^3$, % CaO = 23)

Begin by calculating the sucrose content (S) of the juice using the purity (P) equation:

$$P = \frac{S}{DS} \times 100$$

$$S = \frac{P \times DS}{100} = \frac{88.0 \times 15.0}{100} = 13.2\% \text{ on juice (m/m)}$$

The amount of NS of the juice is

$$NS = DS - S = 15.0 - 13.2 = 1.8\% \text{ on juice}$$

As stated earlier, lime addition should be kept at about 80% CaO on NS. Therefore, the amount of lime required to process this juice is

$$1.8 \times \frac{80}{100} = 1.44\% \text{ on juice}$$

To convert this value into % on beet (OB), multiply the result by 115% (for a typical diffusion draft) and 120% (for a 20% increase in juice between diffusion and limed juice):

$$1.44 \times \frac{115}{100} \times \frac{120}{100} = 2.0\% \text{ OB}$$

Because MOL contains 23% CaO, the amount of MOL is

$$2.0 \times \frac{100}{23} = 8.7\% \text{ OB}$$

The data in the following table are based on the preceding calculations:

Diffusion-Juice Purity	CaO Required (% OB)
90	1.7
89	1.8
88	2.0
87	2.2
86	2.3
85	2.4
84	2.5

The amount of removable nonsugars (RNS) during purification is about one-third to one-sixth the total nonsugar content of the diffusion juice. Therefore, it seems that there is an excess amount of lime equal to about two-thirds to five-sixths. However, it is important to know that the excess of lime is required to produce more $CaCO_3$, which is used as

- Adsorbent for adsorption (sticking of a substance to another based on adsorption capacity)
- Filter-aid for the filtration process to improve the filterability of the juice

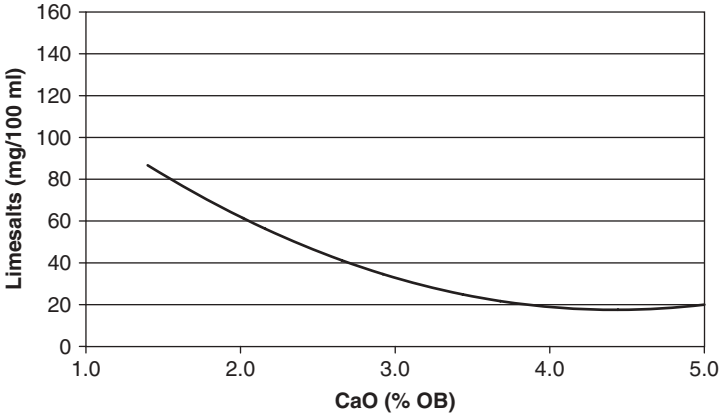


Figure 3.10.2 Effect of lime use on thin-juice limesalts and color

Figure 3.10.2 shows that at normal alkalinities of the first carb (0.08 to 0.10% CaO), the greater the lime use, the lower are the limesalt content and the color of a typical thin juice (McGinnis 1982). However, this is true up to a certain point and must be determined from juice-quality improvement and the amount of lime used for each factory. In general, moderate amount of lime should be used for two main reasons:

- Lime production is expensive
- The sale or disposal of carbonation-lime residue (limecake) is a potential problem

In the sugar laboratory, two important tests are conducted to control the purification process: alkalinity and pH. The alkalinity test gives the quantity of alkali present in the juice. The pH test gives information about the intensity of the reaction, which influences the rates of sucrose inversion, sedimentation, filtration, and decolorization of the juice. Before we get into the details of purification, we need to know something about three related subjects:

- **Alkalinity:** Knowing about alkalinity helps because the juice-purification station, particularly during the first- and second-carbonation steps, is controlled and optimized by measuring the alkalinity and pH of the juice.
- **Hardness** (limesalts): Knowing about limesalts helps because an improper purification produces juice with high limesalts. Such a juice creates difficulties in the next processing stations, particularly in evaporation (because of the formation of **scale** on the evaporator's heating surfaces).
- **Solubility of lime and carbon dioxide:** Knowing about the solubility of lime and CO_2 in the juice helps because these substances do not act until they **dissolve** in the juice. (Note that when we talk about dissolving a substance in a liquid, we talk about its **solubility**.)

ALKALINITY AND HARDNESS

Alkalinity is the total amount of dissolved carbonate (CO_3^{2-}), bicarbonate (HCO_3^-), and hydroxide (OH^-) of Ca, Mg, Na, and K in a solution. In sugar technology, alkalinity is expressed

in g (or mg) CaO per 100 mL of sample. This is known as **%CaO on juice**. It is reported as "**CaO**" because, if carbonate were reported as $CaCO_3$, bicarbonate as $Ca(HCO_3)_2$, and hydroxide as $Ca(OH)_2$, it would create unnecessary complexities. To simplify the expression, the total alkalinity is reported as CaO (see the Measured as CaO subsection in Section 3 of Chapter 1, for more explanations and calculations).

As stated previously, alkalinity is a good indicationf first- and second-carb juice quality. The alkalinity analysis in the sugar laboratory, in fact, allows sugar technologists to operate the purification station based on three important results:

- Alkalinity
- pH
- Hardness (limesalts)

Alkalinity, pH, and hardness of first- and second-carb juice are usually measured by lab analysts on a routine basis. The three types of alkalinity used in the juice-purification process are:

- Natural alkalinity
- Effective alkalinity
- Optimum alkalinity

Natural Alkalinity

Natural alkalinity is the dissolved Na- and K-carbonate content of the juice. The difference between the **alkalinity** (the total of Ca, Mg, Na, and K carbonates, determined by acid titration to be pH = 8.2 or the phenolphthalein endpoint) and **hardness** (the dissolved salts of Ca and Mg, determined by EDTA titration) determines the natural alkalinity:

$$\text{Natural alkalinity} = \text{Alkalinity (titration to pH = 8.2)} - \text{Hardness}$$

NOTE

Both values (alkalinity and hardness) must be calculated in g (or mg) CaO in 100 mL of juice.

Effective Alkalinity

Effective alkalinity is the dissolved Na- and K-carbonate and bicarbonate content of the juice. The determination of effective alkalinity is the same as that of natural alkalinity, but the titration ends at pH = 9.2:

$$\text{Effective alkalinity} = \text{Alkalinity (titration to pH = 9.2)} - \text{Hardness}$$

Sugar technologists determine effective alkalinity to see whether the juice will contain enough alkalinity for further operations. If the alkalinity (refers to titration to pH = 9.2) is larger than the hardness content, the effective alkalinity is a positive number, resulting in normal behavior of the juice during evaporation. If the value is smaller than the hardness content, the effective alkalinity is a negative number, resulting in abnormal behavior of the juice. For example:

- High pH drop during evaporation (0.2 to 0.4 pH drop is usual; anything above 0.4 is unusual)
- High color increase during evaporation (color increase depends also on other factors)
- High pH drop during crystallization

Purified juice with a low effective alkalinity is called **low-buffered juice**. Low-buffered juice decreases in pH and increases in color more than usual during evaporation (a disadvantage), but it does not scale the evaporators rapidly (an advantage). In general, the following are related:

- Color
- Hardness nature
- Hardness content
- Buffering capacity
- Evaporators' boil-out

In the laboratory, the effective alkalinity of first-carb juice is determined (not routinely, but once in a week) to predict the buffering capacity of the thin juice. It is desirable to hold this value between 0.005 and 0.010% on juice (g CaO in 100 mL of juice). If lower, addition of chemicals such as **soda ash** (Na_2CO_3), **caustic soda** (NaOH) or **magnesium oxide** (MgO) is recommended. (Note: There will be more discussion on this topic later in this section under the heading of Addition of Chemicals during Purification Process.)

NOTES

- **Effective alkalinity is a good predictor of juice behavior during further stations (particularly in the evaporation station) because the crystallization operation is at a pH similar to that of the second carbonation (about 9.0).**
- **Sugar technologists use the term buffering capacity to express the alkalinity of the purified juice. Effective alkalinity is a better parameter than natural alkalinity for expressing the amount of buffering content of the thin juice because the titration to pH = 9.2 is closer to the normal pH of second-carb juice (which is about 9.0).**

Optimum Alkalinity

Optimum alkalinity is the alkalinity of the juice after the maximum amount of hardness has been removed. In the laboratory, filtered first-carb juice samples are gassed (addition of carbonation gas) to a different pH (usually to 9.0, 8.9, 8.8, and 8.6), and the hardness of each sample is determined. The sample with optimum alkalinity contains the lowest hardness content. So the pH of the second-carb juice is kept around the optimum.

NOTES

- **In diffusion juice, bicarbonate anions (HCO_3) are present. In limed juice, carbonate (CO_3) and often hydroxide (OH) are present. Usually, the bicarbonate is assumed to be 0 when there is hydroxide. This assumption is not completely valid, but the error is insignificant.**
- **For information about analyses of effective and optimum alkalinity, refer to Chapter 11.**

Hardness

Hardness (limesalts, in the sugar technologist's language) is the amount of soluble salts of calcium (Ca) and magnesium (Mg) in the juice, which is not removed during second carbonation. In the evaporators (due to solubility change), some hardness is deposited on the tubes,

producing **scale**. Calcium carbonate ($CaCO_3$) and calcium oxalate (CaC_2O_4) are the examples of hardness. For example, calcium bicarbonate in the juice precipitates to form calcium carbonate:

$$Ca(HCO_3)_2 + \text{Heat} \rightarrow CaCO_3\downarrow + CO_2 + H_2O$$

The scale decreases heat flow to the juice, which increases the energy use of the factory. In the sugar industry, the hardness is expressed as mg/100 *DS*, which reads "milligrams in 100 grams of dry substance." (Note: Like alkalinity, hardness is reported as CaO.) For example, a juice with 120 mg hardness content means that 100 g dry substance of the juice contains 120 mg of hardness, measured as CaO or 1200 ppm (1200 mg limesalts per one million mg). If the *DS* of the sample is 15%, this equates to $120 \times 15\% = 18$ mg in 100 g juice.

In the laboratory, the amount of hardness in first-carb juice and thin juice is routinely tested to find out the efficiency of the purification process with regard to hardness elimination. In areas where the *hardness content is high* and the *effective alkalinity of the juice is low*, one or more of the following remedies are recommended:

- **Set the alkalinity** of the second-carb juice high to keep the thick-juice alkaline
- **Add chemicals** such as soda ash (Na_2CO_3) or magnesium oxide (MgO) to the juice
- **Install an ion-exchange softening station** to decrease the amount of hardness (see Chapter 6)

The hardness content of thin juice depends on where the sugarbeet is grown. A hardness content of 50 to 200 mg/100 *DS* is normal. Hardness can get too high (up to 600) when a factory processes **damaged beet** (deteriorated beet due to a long campaign period or frost). The hardness content of thin juice is normal in factories in the western part of the United States and many parts of Europe. But in factories in Michigan, Morocco, Spain, and Iran, the thin juice usually contains high amounts of hardness (150 to 600) and low natural alkalinity.

During evaporation, part of the hardness precipitates, forming scale on the evaporators' heating surfaces. But part of hardness passes the evaporation and crystallization process and ends up in molasses. Besides scaling, hardness cause the following problems:

- Increase in the viscosity of the juice when concentrated
- Decrease in the movement of molecules
- Decrease in the crystallization process
- Decrease in sucrose recovery

NOTE

There is always an optimum alkalinity to achieve the lowest hardness (limesalts) content possible in thin juice. Figure 3.10.3 shows the relation between alkalinity and hardness in a typical thin juice.

SOLUBILITY OF CaO AND CO_2

The **solubility** of a **solute** such as CaO, in a **solvent** such as water, is the maximum quantity of the solute that dissolves in a certain quantity of solvent at a specified temperature. Solubility is usually expressed in grams per 100 g solvent (% by mass).

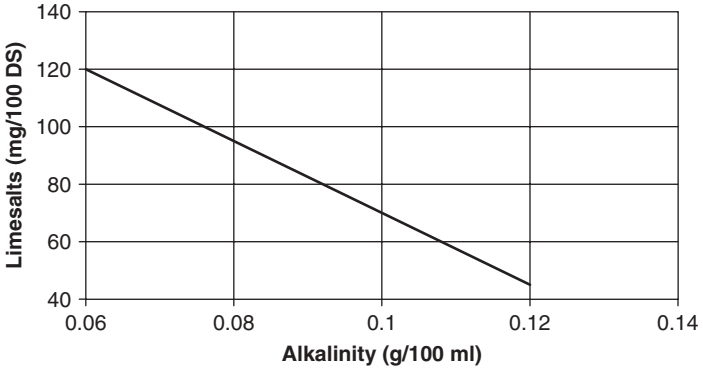


Figure 3.10.3 Effect of alkalinity on limesalts

As stated, complete separation of **removable nonsugars** (RNS) from diffusion juice is the main result of proper juice purification. Essential to this process is the chemical reaction between nonsugars with CaO and CO_2 that results in the elimination of RNS. *Lime and CO_2 cannot act until they dissolve in the juice*, so their solubility is important.

The solubility of CaO and $Ca(OH)_2$ in the juice depends on the following:

- **Temperature:** The solubility of lime decreases with increasing juice temperature. For example, the solubility of CaO in a 14% sucrose solution at 40°C is 1.5% by mass, but at 80°C it is about 0.5%. The reason for the difference in solubility is the higher **saccharate** hydrolysis at a higher temperature.
- **Sucrose concentration:** The solubility of lime increases with increasing sucrose concentration. For example, the solubility of CaO in a 3% sugar solution at 80°C is 0.10 g, but in 14% sugar solution at the same temperature is 0.26% by mass. Note that the greater lime solubility in solutions with higher sucrose concentration is due to the formation of more **calcium saccharate**, $Ca(C_{12}H_{22}O_{11})_2$: Also, note that during the liming process, the amount of CaO used (1 to 3% by mass of the juice) is much higher than can be dissolved (only 0.26% in a 14% sucrose solution). The excess of lime is used as the adsorbent for adsorption of nonsugars and as an aid during the juice-filtration process.
- **Purity of the juice:** The solubility of CaO increases with decreasing juice purity. In other words, the solubility of CaO in impure sucrose solutions is greater than in pure sucrose solutions (at the same sucrose content). For example, CaO is more soluble in diffusion juice than in filtered second-carb juice or in a pure sucrose solution (van der Poel et al. 1998).
- **pH:** The solubility of lime increases if the solution is made more acidic or basic. For example, the presence of NH_3, a base, increases the solubility of CaO. In general, a basic pH condition during the liming process increases the solubility of CaO.
- **Stirring:** The solubility of lime slightly increases when the juice is stirred.

The solubility of CO_2 in the juice depends on the following:

- **Temperature:** The solubility of CO_2 increases with increasing juice temperature. For example, the solubility of CO_2 in a solution containing 12% sucrose and 0.25% citric acid at 10°C is 2.0% m/m, but at 25°C is 1.3%.

- **Pressure:** The solubility of CO_2 increases with increasing pressure. For example, the solubility of CO_2 in water at atmospheric pressure and 25°C is 1.4%, but at 2 atm (2 bar) pressure and the same temperature is 4.1%. Another example is that CO_2 in an unopened bottle of **carbonated beverage** (a soft drink containing CO_2) is dissolved under pressure. Opening the bottle releases the pressure, causing the CO_2 gas to escape out of the solution because it becomes less soluble at lower pressure.
- **Stirring:** The solubility of CO_2 slightly increases when the juice is stirred.

Up to now, you have learned the basics of juice purification. Next, you will learn the main points of the juice-purification process.

CLASSICAL PURIFICATION

Two methods of juice purification are available:

- Classical juice purification
- Defeco-carbonation juice purification

These methods are similar in principle but different in the way the lime and carbon dioxide are added to the juice. The classical method consists of the following four stages:

- Preliming
- Mainliming
- First carbonation
- Second carbonation

In **classical purification**, all four stages are provided independently. In **defeco** (short for defecation) **purification**, the first three stages occur at the same time and in connection with each other (i.e., lime and carbonation gas are added to the juice at the same time). However, to fully understand the defeco-carbonation process, it is helpful to first have a good knowledge of the fundamental operating principles of classical juice purification.

In **classical** (traditional) juice purification, preliming, mainliming, first carbonation, and second carbonation are provided separately. Some factories have a purification station that can operate either in classical mode or in defeco-carbonation mode. These factories usually switch to defeco mode when they process **low-quality beet** (low-purity or damaged beet). Now, the processing steps of the classical method are described in detail, starting with preliming.

Preliming

Preliming is the step of purification where a small amount of lime (about 0.2 to 0.7% on juice) is added to the heated diffusion juice (about 86°C) until optimum conditions for the preliming operation (pH of about 8.5 and an alkalinity of about 0.1) are reached. The liming time of the juice in this step is about 10 to 15 minutes. Preliming has these main functions:

- To neutralize the acidity of the diffusion juice
- To precipitate certain nonsugars
- To destabilize the colloids

The nonsugars that are removed during preliming cannot be removed adequately in mainliming. If preliming is controlled properly, most of the colloids, invert sugar, proteins, phosphates, and sulfates in the diffusion juice are precipitated and removed later by sedimentation and

filtration. In effect, every processing step after preliming depends on the way the preliming is operated.

The way the lime is added to the juice affects the precipitation of the nonsucroses, settling of the juice in the clarifier, its filterability during filtration, and foaming activities. If lime is added at once, known as **full preliming**, the pH increase through the prelimer occurs quickly, and there is not enough time to complete the reactions. As a result, the juice foams excessively, and unsatisfactory settling and filtration occur in later processing steps.

J. Dedek and J. Vasatko, the Czech pioneer sugar technologists, proved that better results are achieved when diffusion juice is limed in portions and in different stages, known as **progressive preliming**. In progressive preliming, the alkalinity of the juice is controlled by a gradual increase in the pH.

As shown in Figure 3.10.4, three types of pH curves are possible in preliming (McGinnis 1982). Only the curve created by progressive preliming (type C) produces the best result. The type C curve is achieved by a small increase in pH in the first steps, followed by larger increases in later steps. As the curve type proceeds toward type A, an incomplete preliming results, which creates excess foaming and other difficulties in sedimentation and filtration.

In general, progressive preliming has the following advantages over full preliming:

- Better juice color
- Better colloid destabilization
- Better juice settling and filterability

Beet-sugar factories use two types of preliming: **hot** (at 80 to 85°C) and **cold** (at 30 to 40°C). Purification systems such as BMA use hot preliming, and some, such as DDS, use cold preliming. **Cold preliming** is more effective because *decreasing temperature increases the solubility of CaO in the juice*. Lime will go completely into solution and not remain as a suspension in the juice. Therefore, the amount of lime used in cold preliming (0.3 to 0.7% CaO on juice) is higher than in hot preliming (0.2 to 0.5% on juice). Following is a comparison of hot and cold preliming:

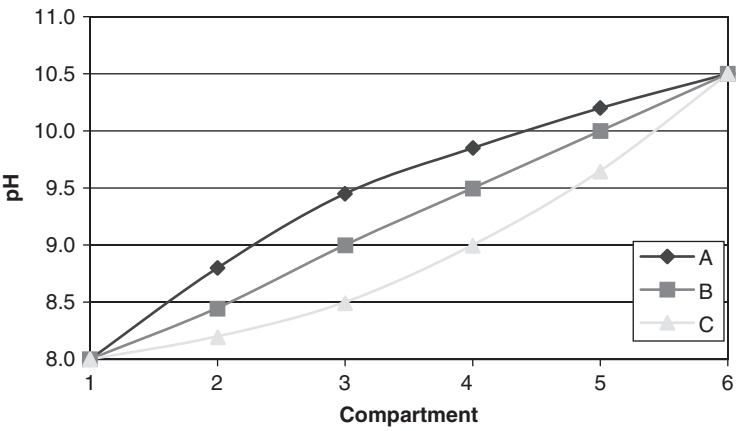


Figure 3.10.4 Prelimer pH curve types

- **Retention time:** Hot preliming requires less time (about 10 minutes compared to about 15 minutes for cold preliming)
- **Juice filterability:** Hot preliming produces juice with better settleability and filterability
- **Juice color:** Hot preliming produces juice with higher color
- **Foam:** Hot preliming produces juice with less foam

The prelimer should be operated such that sufficient time is available for a *gradual increase in pH and alkalinity* throughout the process. Settling and filtration problems and production of high-color juice when processing good beets is generally an indication of improper preliming. Proper preliming provides the following results:

- Increase in the efficiency of filtration and sedimentation of the juice
- Increase in the purity of the juice by 0.3 to 0.5%
- Increase in the pH value to about 8.5
- Decrease in lime usage
- Decrease in juice color during evaporation
- Decrease in the hardness content of the juice
- Decrease in proteins, pectins, and other colloidal substances of the juice

Next, on the **chemistry of liming**, we need to know that reactions occurring during liming are divided into two groups:

- Reaction of lime with nonsucroses
- Reaction of lime with sucrose

The first group of reaction can be subdivided into four:

- Coagulation
- Precipitation
- Neutralization
- Decomposition

Coagulation reactions are the most important functions of liming because diffusion juice contains colloids. Again, colloids cause major difficulties in sedimentation and filtration because of their high molecular mass. Examples of colloidal substances, or simply **colloids,** are pectin, dextran, colorants (coloring substances), organic compounds from decaying beet particles, clays, foam, and microorganisms. Colloids are

- Sticky
- Viscose
- Not settleable
- Negatively charged
- Long-chain polymers
- Not dissolved in water

Colloids (in Greek means *glue*) are large molecules (1 to 100 nm, 1 nm = 10^{-9} m) but small enough to remain suspended (do not settle on standing). The molecular mass of colloids may

be viewed as representing its chain length (*the greater the molecular mass, the greater is the chain length*). During filtration, long-chain colloids stick to the surfaces of the filter media and instantly plug their pores. Lime can **coagulate** (neutralize and destabilize) the colloids. During preliming, the lime, which has a positive charge, combines with negative sites on the colloid to produce colloid calcium carbonate (CCC), which is neutral in terms of charge. The CCC can be viewed as a ball covered with lime that is no longer sticky to create difficulties during filtration. The process of changing the colloids to CCC is called **colloid coagulation**.

Precipitation reactions are the reactions of Ca from lime with certain nonsucrose salts to precipitate calcium salts. For example, the lime reacts with sodium or potassium oxalate to precipitate calcium oxalate (CaC_2O_4), which is removed in the first-carb filtration:

$$Na_2C_2O_4 + Ca(OH)_2 \rightarrow CaC_2O_4\downarrow + 2NaOH$$

$$K_2C_2O_4 + Ca(OH)_2 \rightarrow CaC_2O_4\downarrow + 2KOH$$

(The ↓ represents precipitation.) After liming, more alkaline hydroxides (NaOH and KOH) are present in the juice than $Ca(OH)_2$. Then the sodium and potassium hydroxides react with CO_2 during carbonation to form alkaline carbonates, which increase the solubility of the CO_2 in the second carbonation:

$$2NaOH + CO_2 \rightarrow Na_2CO_3 + H_2O$$

$$2KOH + CO_2 \rightarrow K_2CO_3 + H_2O$$

Neutralization reactions are the reactions between free acids in the diffusion juice with lime. The reactions of **oxalic acid** ($H_2C_2O_4$), **citric acid** ($H_3C_6H_5O_7$), and **tartaric acid** ($H_2C_4H_4O_6$) with lime to neutralize their acidity are the best examples of this type of reaction. The diffusion juice has a pH value of 6.0 to 6.5 (diffusion juice acidity is equal to about 0.04% CaO) and is not suitable for further operation. For example, oxalic acid reacts with lime to precipitate (↓) neutral **calcium oxalate** (CaC_2O_4):

$$H_2C_2O_4 + Ca(OH)_2 \rightarrow CaC_2O_4\downarrow + 2H_2O$$

Decomposition reactions are the reactions between lime and substances such as invert sugar, ammonium salts, and glutamine (one type of amino acids). The products of these reactions either *are not precipitates* or are *precipitates but easily filterable*. For example, the products of the decomposition of invert sugar are acids and coloring materials, and the products of beet fat decomposition are glycerin and fatty acids. The glycerin remains in solution, and fatty acids precipitate with lime to their calcium salts (Silin 1964).

The products of decomposition of glutamine is the pyrollidone carboxylic acid (PCA) and ammonia. Then the PCA reacts with lime and is removed later with filtration. Ammonia (a base substance) remains in the juice during purification (because it is stable in alkaline solutions) but eventually escapes from the juice during evaporation (*one of the causes of pH drop during evaporation*).

Next, we consider the reaction of lime with sucrose, or the chemistry of the **water-sucrose-lime** ($H_2O–C_{12}H_{22}O_{11}–CaO$) system that forms when lime is added to the juice. To make the description easier, we will disregard the presence of nonsucroses, since their influence on this system is insignificant. The chemical reactions in this system can be expressed by the following equation:

$$Ca(OH)_2 + C_{12}H_{22}O_{11} \rightarrow Ca(C_{12}H_{22}O_{11})_2 + 2H_2O$$

Previously, we learned of the solubility of CaO in the juice and stated that during the liming process, the amount of CaO mixed with the juice is much higher than can be dissolved in the juice, so some free lime stays in the juice. After lime is added, sucrose is precipitated in the form of saccharate. As a result, a new system containing **water-sucrose-lime-saccharate** [H_2O–$C_{12}H_{22}O_{11}$–CaO–$Ca(C_{12}H_{22}O_{11})_2$] is formed, which remains until the start of carbonation (adding CO_2 to the juice).

An important point is that only a small amount of sucrose in the juice reacts with lime and becomes **calcium saccharate** ($Ca(C_{12}H_{22}O_{11})_2$). This conversion varies with temperature. The next example shows how much sucrose reacts with lime at temperatures similar to preliming (around 80°C).

EXAMPLE 3

Given:

Solubility of CaO in a 14.0% sucrose solution at 80°C	0.26% by mass
Solubility of CaO in pure water at 80°C	0.07%

Calculate:

Percentage of sucrose converted into saccharate

Because it may be assumed that the amount of free lime in a solution that contains 14.0% sucrose and 0.26% CaO is also 0.07%, the lime in the form of saccharate in this solution can be calculated as follows:

$$0.26 - 0.07 = 0.19\%$$

Molecular mass (*MM*) of sucrose is 342.3 g/mole and equivalent mass (*Eq*) of CaO 56.0/2 = 28.0. The amount of sucrose saccharate formed by 0.19% CaO is

$$\frac{342.3 \times 0.19}{28.0} = 2.3\%$$

The percentage of sucrose saccharate is

$$\frac{2.3}{14.0} \times 100 = 16.4\%$$

Therefore, the percentage of free sucrose will be

$$100 - 16.4 = 83.6\%$$

Two basic types of **prelimers are** used in sugar factories:

- Horizontal
- Vertical

The following explanation should be considered general because prelimer designs vary among different manufacturers.

A **horizontal prelimer** (B-M prelimer, from its inventors, Breieghel and Muller) consists of a U-shaped vessel about 10 m (30 ft) long and 5 m (15 ft) wide divided into six or seven compartments by partitions. (See Figure 3.10.5.) To allow movement of the juice, the partitions do not extend all the way to the bottom. A shaft with stirring paddles rotates in the lower part to mix the juice. A **baffle** (deflector) is fixed on the top part of each partition. The baffles can be adjusted to different angles.

The prelimer works in countercurrent mode such that diffusion juice moves by gravity from one end to the other, while the milk-of-lime (MOL) is delivered into the last compartment, from which the juice is discharged. MOL is propelled toward the juice by the influence of the baffles. The inclined baffles push part of the juice-lime mixture back toward the juice. Therefore, by adjusting the angle of the baffles and the speed of the agitator, the correct progressive distribution of lime concentration, from zero at the one end to highest at the other end, can be achieved. For example, a typical alkalinity (in g CaO in 100 mL of juice) of mixed juice in a prelimer with seven compartments is as follows:

Compartments	1	2	3	4	5	6	7
Alkalinity	0.01	0.02	0.04	0.08	0.12	0.18	0.24

A **vertical prelimer** consists of a cylindrical tank separated into seven compartments, with a shaft mounted to stirring paddles in each compartment for mixing the juice. (See Figure 3.10.6.) To allow the flow of mixture, the plates separating the compartments do not quite reach the side. The prelimer works in countercurrent mode; juice enters at the bottom and flows upward, and **MOL** enters at the top and flows downward.

Vertical prelimers require less room and their mixing process is more effective. So they are gaining in popularity. In general, the prelimer operation depends on the following variables:

- Amount of lime used
- Equipment adjustments
- Amount of recycled mud
- Entering points of recycled mud

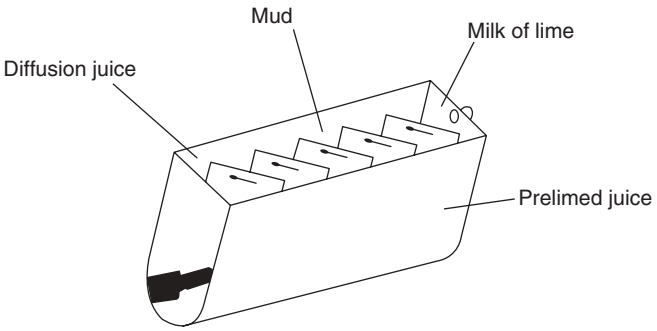


Figure 3.10.5 Horizontal prelimer

EXAMPLE 4

Given:

Diffusion juice *DS*	14.0%
Capacity of the factory	8000 t/day
Draft	115%
Prelimer retention time	10 min
Preliming temperature	80°C

Calculate:
Capacity of a prelimer to handle the preliming operation

The amount of diffusion juice ($m_{\text{Dif. J}}$) produced is

$$m_{Dif.J.} = \frac{8000 \times 1000}{24 \times 60} \times 1.15 = 6389\,\text{kg/min}$$

At 14% *DS* and 80°C, the density of the juice is about 1.03 kg/L and the volume of the juice will be

$$v = \frac{m}{d} = \frac{6389}{1.03} = 6203\,\text{L/min}$$

At 10 minutes holding time, 10% increase in juice between diffusion and prelimed juice, and 10% extra capacity for foam, the required capacity of the prelimer is

$$\frac{6203}{1000} \times 10 \times 1.10 \times 1.10 = 75\,\text{m}^3\,(2627\,\text{ft}^3)$$

Mainliming

Mainliming (main defecation) is the step of purification where lime (at about 1.0 to 2.5% OB) is added to the heated prelimed juice (about 88°C). Mainliming is performed for the following good reasons:

- To increase the pH and alkalinity of the juice
- To complete the reactions between nonsugars and lime
- To overlime the juice (the excess lime is used as filter aid)

Unlike preliming, in mainliming the lime used is much more than can be dissolved in the juice for the following reasons:

- To act as an adsorbent for the **adsorption** of nonsugars (lime has a high adsorption capacity)
- To act as an aid for the filtration process to improve the filterability of the juice

The most important reactions occurring during mainliming are the following:

- Decomposition of *invert sugar* into colorants and acids and the formation of oxalic acid
- Decomposition of *amino acids* such as glutamine and asparagine into their ammonium salts

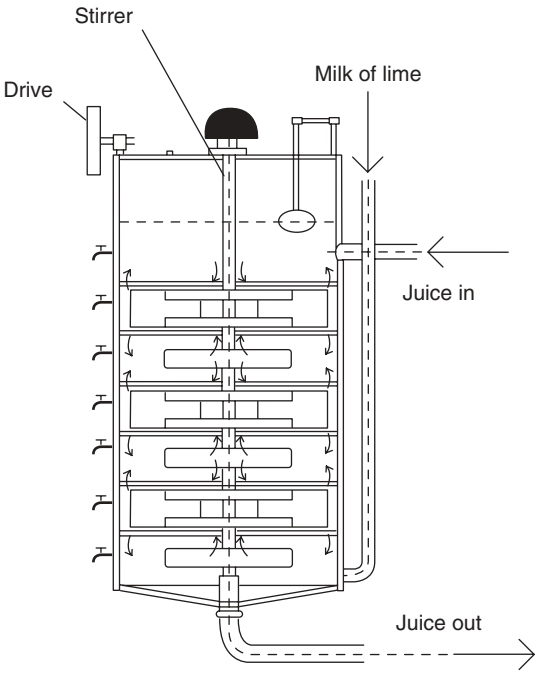


Figure 3.10.6 Vertical prelimer

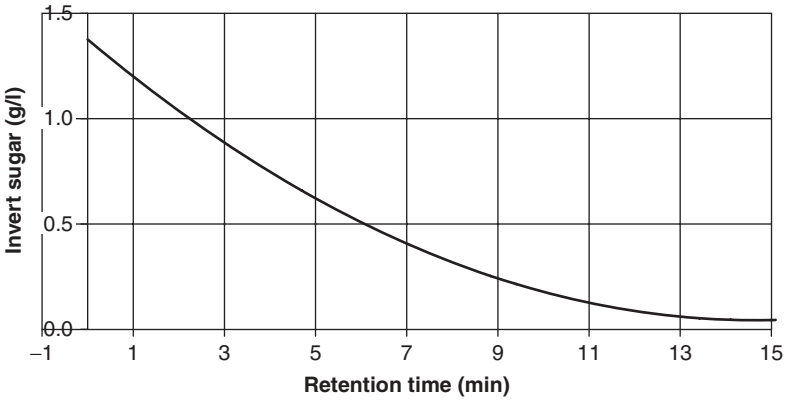


Figure 3.10.7 Invert sugar decomposition as a function of retention time

Figure 3.10.7 shows the effect of retention time on the decomposition of invert sugar during mainliming at 12.5 pH, 85°C, and at moderate liming of 1% CaO on juice (van der Poel et al. 1998). The final point (invert sugar = 0.02 g/L) is considered optimum.

Glutamine accounts for about half the amino acid content of the beet juice. During purification, glutamine is decomposed mainly to pyrrolydone-carboxylic acid (PCA) and to less

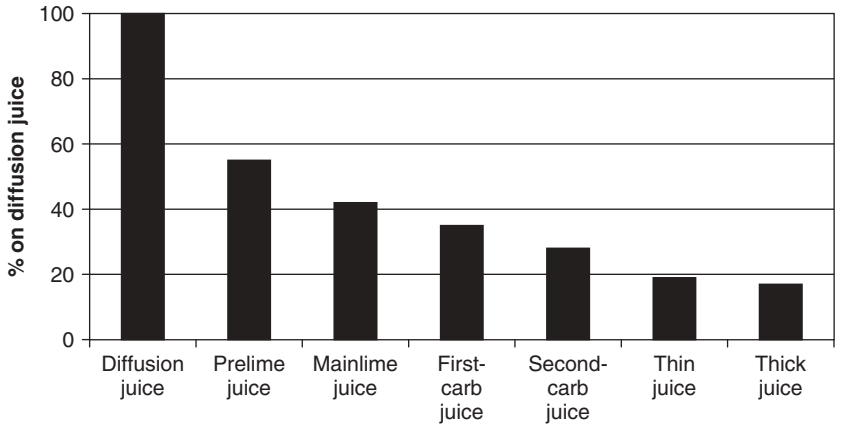


Figure 3.10.8 Glutamine content in in-process products as % on diffusion juice

extent to glutamic acid. The PCA causes a pH drop later in the process (mainly during evaporation). The degree of glutamine decomposition depends on the purification operation. However, some glutamine stays with the juice through the entire operation and ends up in molasses. Figure 3.10.8 compares the typical glutamine content in different in-process products (juices, syrups, and liquors) with that in diffusion juice.

NOTES

- **Higher purity, better thermostability (stable to heat), and less-color-juice are among the main color-juice advantages of mainliming. Produced acid reacts with lime to form hardness (limesalts), which is a disadvantage of mainliming.**
- **A large part of the limesalts produced during purification is adsorbed by the calcium carbonate precipitate, leaving relatively low limesalts in the juice after filtration.**

First Carbonation

First carbonation (see Figure 3.10.9) is the step of purification where the carbonation gas is added to the heated limed juice (about 90°C) until the optimum conditions for the first-carb juice (pH of 10.8 to 11.0 and alkalinity of 0.08 to 0.11) are reached. The gassing time of the juice in this step is about 10 minutes.

After the first-carbonation process, the juice becomes slurry, named **first-carbonation slurry**. In the juice are suspended solids visible to the naked eye as individual substances. Carbonation slurry consists of calcium carbonate (55 to 60%), organic compounds (10 to 15%), and water. To separate the solids from the juice, the first-carbonation slurry is treated in **clarifiers** (settlers) or **thickening filters**. The separation of the solids converts the first-carb slurry into **clarified juice**, which is nearly free of suspended solids, and a thickened product called **carbonation mud**. In purification systems equipped with **carbonation-mud recycling**, 50 to 60% of the mud is pumped back to the prelimer. The rest of the mud is filtered by **cake filters**



Figure 3.10.9 Carbonation tanks

(rotary-drum filters or filter presses) to produce the wet filter cake residue, called **carbonation-lime residue** or **limecake** (a by-product of the beet-sugar factory). (Note: In this book, the following three terms are used for expression of limed-juice products in different stages:)

- **Carbonation slurry:** The slurry produced after the first-carb juice is limed (actually limed juice)
- **Carbonation mud:** The solids separated from the first-carb slurry in the clarifier (or filter)
- **Carbonation-lime residue** (CLR or lime residue)**:** The wet cake residue produced from further concentration of carbonation mud in cake filters

The first carbonation has three main functions:

- To release sucrose from saccharate carbonate
- To react with unreacted lime to precipitate calcium carbonate
- To react with dissolved lime to precipitate calcium carbonate

Sucrose is released from saccharate according to the following equation:

$$C_{12}H_{22}O_{11}.CaO + CO_2 \rightarrow C_{12}H_{22}O_{11} + CaCO_3\downarrow$$

(Note: The reaction between calcium saccharate ($C_{12}H_{22}O_{11}.CaO$) and lime is more complicated than the preceding simple formula.) Some sugar scientists call this reaction **calcium sucrocarbonate** and express it as $(C_{12}H_{22}O_{11})_X.(CaCO_3)_Y.(CaO)_Z$. To achieve the preceding reaction, CO_2 must be dissolved in water to produce carbonic acid:

$$CO_2 + H_2O \leftrightarrow H_2CO_3$$

Then the carbonic acid reacts with the dissolved lime and precipitates calcium carbonate:

$$H_2CO_3 + Ca(OH)_2 \rightarrow CaCO_3\downarrow + 2H_2O$$

The precipitated calcium carbonate (PCC) acts in the following important ways:

- **Adsorption of nonsucroses:** The adsorption of nonsugars by PCC is due to the positive charge of $CaCO_3$, which can adsorb substances with negative charges, such as colloids. Another reason for adsorption is the difference in **surface tension** (the elastic tendency found at the surface of a substance) between nonsucroses and PCC. Some nonsugars (e.g., hardness and coloring materials) have a high surface tension, so they can be adsorbed by calcium carbonate and easily separated later by sedimentation (in the clarifier) or filtration. As a result, the purity of the juice increases. (Note: The lime that could not react with H_2CO_3 remains in the juice even after carbonation and becomes part of the hardness.)
- **Acting as a filter aid:** Excessive PCC works as a filter aid during sedimentation and filtration of the juice. As was stated, although the amount of nonsugars of diffusion juice that can be precipitated with lime (removable nonsugars, RNS) is about 0.5% OB, during purification 1.5 to 2.5% CaO on beet is used. This means that the amount of CaO is threefold to fivefold. The excess is used as a filter aid.
- **Acting as a reagent:** PCC acts as a reagent for the further purification of the juice.

The operation of first carbonation is a compromise because conditions that usually improve its operation can worsen the sedimentation and filtration operations. For example, the operation of first carb at a higher alkalinity produces juice with low color and hardness (advantage) but decreases the rate of the sedimentation of the first-carb juice in the clarifier (disadvantage). In general, any condition that increases the surface of PCC (e.g., decreasing the particle size) improves the operation of first carbonation (*the smaller the particle size of PCC, the larger is the surface area and the higher is the purification effect*). This is a negative point for the filtration process (*the smaller the particle size of PCC, the lower is the filtration rate*). As a result, one can state that the ideal situation is to maintain the best possible purification conditions without creating difficulties for the settling and filtration operations.

The quality of the first-carb operation depends on the following:

- **Lime use:** The amount of lime used during the liming process greatly affects the process.
- **Temperature:** At low temperatures, the precipitated calcium carbonate (PCC) particles are small (with a high surface area), which is an advantage for better adsorption of nonsugars but a disadvantage for the filtration process. In addition, juice creates a lot of foam. The optimum temperature for the first-carb operation is between 80 and 85°C.
- **Alkalinity:** If the juice is overcarbonated (to an alkalinity below 0.07), it has a darker color and more hardness. On the other hand, if the juice does not get enough gas and is undercarbonated (to an alkalinity over 0.12), it contains calcium saccharate, which is difficult to

filter and results in high sugar losses. Filtration problems occur because the juice becomes slimy and gelatinous rather than crystalline. The optimum alkalinity of first carbonation is between 0.08 and 0.11, which is equal to pH of 10.8 to 11.1.

- **Rate of carbonation gas utilization** (R_{CG})**:** This is the percentage of CO_2 in the carbonation gas entering the carbonation tank $(CO_2)_{In}$ to CO_2 in the gas leaving the tank $(CO_2)_{Out}$, in other words, the percentage of gas **absorption** by the juice. It can be calculated by the following formula:

$$R_{CG} = \frac{(CO_2)_{In} - (CO_2)_{Out}}{(CO_2)_{In} \times [100 - (CO_2)_{Out}]} \times 10000 \qquad (3.10.1)$$

The rate of gas utilization of the first carbonation is usually between 75 and 85%, and the R_{CG} of the second carb is between 60 and 70%.

EXAMPLE 5

Given:

CO_2 content in	33.2%
CO_2 content out	8.5%

Calculate:
CO_2 utilization

$$R_{CG} = \frac{33.2 - 8.5}{33.2(100 - 8.5)} \times 10000 = 81.3\%$$

The following are important criteria for first-carb operation:

- pH (10.8 to 11.0)
- Time (10 to 12 min)
- Temperature (85 to 90°C)
- Alkalinity (0.07 to 0.1% CaO)

Some factories add **lime to the filtered first-carb juice** in a small amount (about 0.2 to 0.4% OB) before the second carbonation. This is the practice particularly when the first-carbonation process is left unfinished or when damaged beets are processed. Benefits derived from adding the lime are as follows:

- Decrease in color
- Decrease in limesalts
- Decrease in nonsugars
- Increase in juice filterability

First-carbonation operations are controlled by pH measurements, the sedimentation rate, and frequent alkalinity titration (see Chapter 11 for methods of analyses). The optimum sedimentation rate for the best settling of a first-carb juice, produced from normal quality beets, is 4 to 6 cm/min (1.6 to 2.4 in./min). To determine the sedimentation (settling) rate, a simple test is

usually carried out by the purification operator. The procedure for running this test is explained in Section 11 of Chapter 3.

First-carb juice separated from mud in the clarifier or thickening filters is filtered to remove the remainder of solid particles in **pressure-leaf filters** (see Section 11 of Chapter 3) because these solids would otherwise partly redissolve in the **second carbonation**.

Second Carbonation

Second carbonation is the step of purification where carbonation gas is added to the filtered and heated first-carb juice until optimum conditions for the second-carb juice (pH of 9.0 to 9.2 and alkalinity of 0.02 to 0.03) are reached. Before the filtered first-carb juice is entered into the second-carb tank, it is heated to about 92°C in the second-carb heaters to prevent formation of calcium bicarbonate in the juice. The main functions of the second carbonation are:

- To precipitate still-existing unreacted lime
- To decrease the hardness content of the juice to minimum
- To decrease the pH of the juice to an optimum level for the next station

The following are important criteria for second-carb operation:

- **Temperature:** The juice should be heated to a high temperature (90 to 95°C) before entering the second-carb tank because calcium bicarbonate decomposes to calcium carbonate at temperatures above 90°C (during juice heating and evaporation):

$$Ca(HCO_3)_2 \leftrightarrow CaCO_3\downarrow + CO_2 + H_2O$$

- **Alkalinity:** 0.025 to 0.03% CaO
- **pH:** 8.8 to 9.0

Optimum gassing of the second-carb juice is important. For example, undergassed juice typically contains a higher hardness because the calcium carbonate converts to soluble calcium hydroxide at pH above 10. However, the filtered juice after second carbonation (thin juice) always contains a certain amount of hardness. A hardness content of 50 to 200 mg/100 *DS* is normal, while hardness is higher if the factory processes damaged beets because of a long campaign period or frost. In general, the hardness content in thin juice depends mainly on where the sugarbeet is grown.

If the second carbonation is not performed on the juice, the following problems will occur:

- Scaling of the evaporators tubes
- High molasses production and high sugar loss

Filtered first-carb juice contains $CaCO_3$, $Ca(OH)_2$, alkaline hydroxides (mainly, KOH and NaOH), dissolved limesalts (dissolved salts of calcium and magnesium), and sucrose. The addition of CO_2 creates the following reactions:

$$CO_2 + H_2O \rightarrow H_2CO_3$$

$$CaCO_3 + H_2CO_3 \rightarrow Ca(HCO_3)_2$$

$$Ca(OH)_2 + H_2CO_3 \rightarrow CaCO_3 + H_2O$$

$$2KOH + H_2CO_3 \rightarrow K_2CO_3 + 2H_2O$$

$$2NaOH + H_2CO_3 \rightarrow Na_2CO_3 + 2H_2O$$

A juice heater, a gassing and holding tank, and second-carb filters are the main equipment for the second-carb process. The second-carb tank is similar to the first-carb unit. After second carbonation, the juice is filtered in second-carb filters (pressure-leaf filters). (Note: In some factories, the filtered juice is filtered once more in **safety filters** to ensure that no solid particles are left in the juice.)

DEFECO-CARBONATION PURIFICATION

Defeco is an abbreviation for defecation, which is used in sugar technology to express the action of liming and gassing the juice at the same time. In the **defeco-carbonation method** (liming-gassing method), unlike the classical purification method, both the **liming** (adding lime to the juice) and **carbonation** (adding CO_2 to the juice) are carried out simultaneously. The lime is added to the juice progressively (at a slow and steady rate) and carbonation gas is applied continuously. Some defeco-carbonation systems (e.g., the BMA system) have a separate prelimer. In other systems (e.g., the Dorr system), all three processes of preliming, mainliming, and first carbonation occur in one unit.

The defeco method has the following advantages over the classical method (preliming/mainliming method):

- Processes beets of varying quality (including low-purity and damaged beets) efficiently
- Produces a precipitate with better settleability, filterability, and absorbability
- Has a lower equipment cost and requires less space
- Produces a higher purity juice

NOTE

Some sugar factories with classical purification have equipped their first-carb tanks with lime addition and juice recirculation equipment. In these factories, preliming and mainliming can be omitted, if necessary, and the process changed to defeco-carbonation.

The following are the two main differences between defeco and classical purification:

- In the defeco method, a large **recirculation** (up to 700% on incoming juice) of limed-carbonated juice takes place. This means that during defeco-carbonation, the juice is pumped continuously back and forth to the same tank (if using the Benning carbonator) or between two tanks.
- All defeco-carbonation systems are equipped with first-carb mud-separation equipment (a clarifier or a filter thickener) for separating and thickening the precipitate from the juice. Part of the thick mud (also called slurry or sludge) is returned to the prelimer.

The recirculation of first-carb juice is known as **first-carb juice recirculation**. The returning part of thickened carbonation mud to the prelimer is called **carbonation-mud recycling**. Both actions are explained next.

First-Carb Juice Recirculation

In all defeco systems, the entire carbonated (gassed) juice is **recirculated** during the first-carbonation process. The standard recirculation rate is 700% (7:1) and can be varied from 5:1 to 10:1. Recirculation has the following benefits:

- Prevents foaming
- Improves CO_2 utilization
- Improves settling of the mud in the clarifier

First carbonation is usually carried out in two tanks: A (gassing tank) and B (liming tank). Carbonation is performed in tank A; and lime is added to tank B, and the recirculation is performed between both tanks (see Figure 3.10.10).

A recirculation of 7:1 means that for every ton of juice added to the carbonation tank, 7 tons of carb juice is sent (by pump or natural circulation) from the liming tank back to the gassing tank. (Note: This is the ratio [on juice] of material entering the first-carb tank, and not the juice entering the purification station.) The volume of the first-carb juice differs from the juice entering the purification station by the amount of additions such as lime, melter returns, excess sweet water, and beet-end **sump** (a pit in the ground for collecting waste juices and pumping them back to the process). The recirculation rate (*RR*) can be calculated by the following equation (see Figure 3.10.10 for numbers referring to juice in and out):

$$RR = \frac{(CaO)_2 - (CaO)_1}{(CaO)_3 - (CaO)_2} \times 100 \qquad (3.10.2)$$

EXAMPLE 6

Given:

CaO of precarb juice (point 1)	0.34%
CaO of juice leaving gassing tank (point 2)	0.84%
CaO of juice leaving liming tank (point 3)	0.91%

Calculate:

RR between the gassing tank and the liming tank

$$RR = \frac{0.84 - 0.34}{0.91 - 0.84} \times 100 = 714\%$$

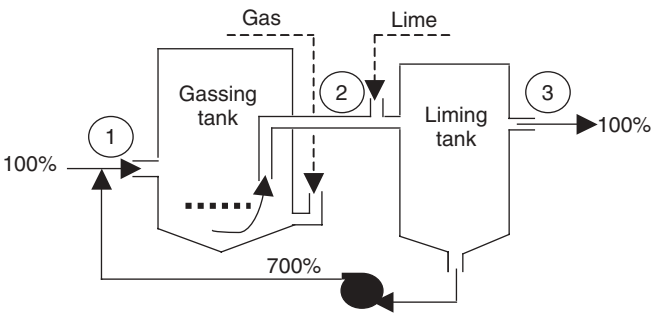


Figure 3.10.10 First-carbonated-juice recirculation

A valid question here is why such a high recirculation rate is required. This question can be answered by the next example.

EXAMPLE 7

Given:

Alkalinity of the juice entering precarb tank (point 1)	0.08% CaO on juice
Alkalinity of the juice leaving liming tank (point 2)	0.09%
Recirculation rate (*RR*)	800%

Calculate:
Alkalinity of first-carb juice

800 mL of recycled juice contains

$$8 \times 0.09 = 0.72\%\ \text{CaO}$$

The alkalinity of first-carb juice distributed in 900 mL of juice (800 recycled juice and 100 mL incoming juice) is

$$\frac{0.72 + 0.08}{8} = 0.1\%\ \text{CaO}$$

This alkalinity is similar to the alkalinity required for first-carb juice (0.08 to 0.11 gCaO/100 mL), which would not be achieved if such a high recirculation did not exist.

Carbonation-Mud Recycling

In all defeco-purification systems, part (50 to 60%) of the **first-carb mud** (the solids separated from the first-carbonation slurry in the clarifier or thickening filters) is returned into the prelimer as a recycle flow. The rest of the carbonation mud goes to the **cake filters** (rotary drum filters or filter presses) for further concentration.

First-carb mud contains high amounts of calcium carbonate. Some factories return mud from first and second carbonation to the prelimer. When the mud is recycled, the prelimed juice is mixed with several volumes of a juice with a much higher pH (mud has pH of 10.5 to 11.0). Mixing mud with diffusion juice has the following benefits:

- **Decreases lime usage:** Mud covers part of the fresh lime
- **Increases coagulation ability:** Mud improves the coagulation of the colloids
- **Increases absorption ability:** Lime in the mud acts as seed to increase the absorbing power of nonsugars
- **Increases** pH **and alkalinity:** Mud increases the diffusion juice's pH to about 8.5 and its alkalinity to 0.08, which are optimum for precarbonation operations
- **Decreases energy cost**

Two types of equipment are used for the separation of carbonation mud from first-carb juice:

- Clarifiers
- Filter thickeners

A typical thickened, first-carbonation mud separated by a clarifier has a concentration of 36% *DS* (about 20 Baume [Be]) with about 18% CaO content. The amount of recycled mud to the prelimer depends on the concentration of the product, and it usually ranges from 50 to 60% of the total mud, which equates to 3 to 6% on diffusion juice. The amount of **recycled-carbonation mud** (*RCM*) on diffusion juice can be calculated from the following equation:

$$RCM = \frac{A}{B} \times 100 \tag{3.10.3}$$

A	%CaO in prelimed juice
B	%CaO in recycled-carbonation mud

EXAMPLE 8

Given:

Amount of CaO in prelimed juice (A)	0.6%
Amount of CaO in recycled carbonation mud (B)	19.0%

Calculate:

Amount of recycled-carbonation mud (*RCM*) to the prelimer

$$RCM = \frac{A}{B} \times 100 = \frac{0.6}{19.0} \times 100 = 3.2\% \quad \text{on diffusion juice}$$

PURIFICATION SYSTEMS

Several purification systems (such as BMA, DDS, DORR, and Putsch) use the defeco technique. Here, three common systems are explained:

- BMA system (used all over the world)
- DDS system (used mostly in Europe)
- Dorr system (used mostly in the United States)

BMA Purification System

The Braunschweigische Maschinenbau Anstalt (BMA) purification system consists of hot preliming, mainliming, first carbonation, clarification (BMA systems uses **clarifiers**), cake filtration, and second carbonation. In the current defeco system, BMA-65, preliming is replaced by the following three steps:

- Juice mixing
- Precarbonation
- Intermediate liming

This type of preliming operates differently than classical preliming because it gets both milk of lime (MOL) and carbonation gas at the same time. In the BMA system, a portion of the limed juice is recirculated from the bottom of the intermediate (liming) tank to the bottom of the precarbonation (gassing) tank. The recirculation rate between the two tanks is kept at about 700% on juice entering the precarbonation tank (see Figure 3.10.11).

The preliming is controlled by the same rules as those of classical preliming. Therefore, a similar normal pH curve (type C; see Figure 3.10.4) is required for preliming. The mixed-juice tank can be considered the first compartment, the precarbonation tank, the middle, and the intermediate tank, the last compartment of the horizontal prelimer. MOL is usually added to the intermediate and mainliming tanks. Sometimes, particularly when damaged beets are processed, MOL is also added to the filtered-first-carb juice before second carbonation.

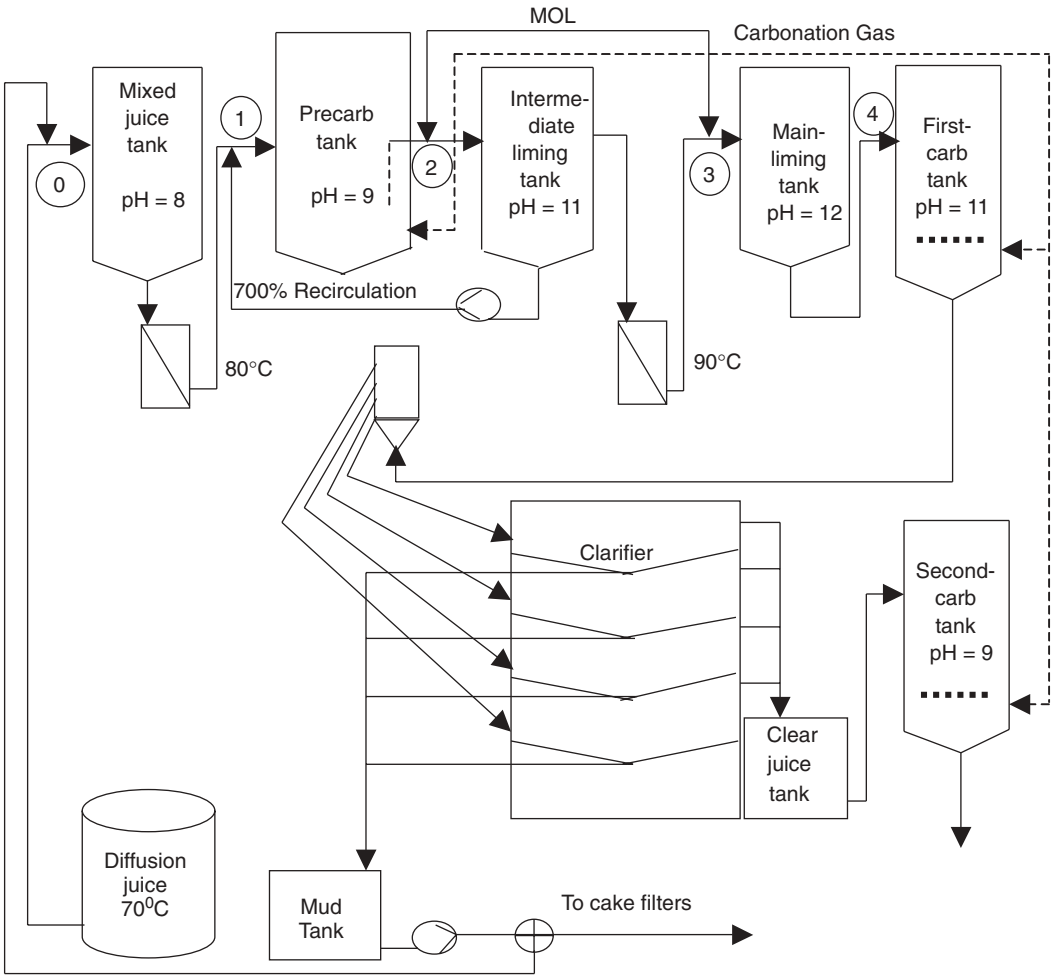


Figure 3.10.11 BMA juice-purification system

The following are the highlights of the operation of the BMA purification system:

1. Diffusion juice is heated to about 70°C.
2. The heated juice enters the mixed-juice tank along with part of the first-carb mud (recycled from the clarifier) to increase the juice pH to about 8.0 (equal to about 0.6 alkalinity). The effluent from the mixed-juice tank is heated to about 80°C.
3. The heated juice is introduced to the precarbonation tank. The pH of the juice in the precarb tank is kept at about 9.0 by the addition of carbonation gas. The precarb tank is connected to the intermediate-liming tank. A portion of the limed juice is recirculated from the bottom of the intermediate tank to the bottom of the precarb tank.
4. The limed juice from the top of the precarb tank is introduced to the intermediate-liming tank. At the point where the juice enters the intermediate tank, approximately 40% of the total MOL is added. The recirculation rate between the intermediate and precarb tanks is kept at about 700% on juice entering the precarb tank. The pH of the recycled juice is kept at about 11.0 by controlling the recirculation-pumping rate.
5. The juice exiting the intermediate tank is heated to about 90°C.
6. The heated juice is pumped to the mainliming tank, where the balance of MOL (about 60%) is added. The pH of the juice leaving the mainliming tank should be kept slightly higher than endpoint of first carbonation (about 12.0 pH).
7. The juice is pumped to the first-carb tank where carbonation gas is introduced in a countercurrent way. The juice is held at the optimum pH of the first carb (about 11.0 pH).
8. The juice is then clarified in a clarifier for mud separation. Part of the underflow from the clarifier (50 to 60%) returns to the mixed-juice tank. The rest of the underflow goes to the **cake filters** (rotary drum filters or filter presses) for further concentration. Filtrate from the cake filters is filtered with the first-carb filters.
9. The clear juice (clarifier's top zone) and filtrate from the first-carb filters are pumped to the second-carb tank, where they are gassed to an alkalinity of 0.02 to 0.025 (pH of 9.0 to 9.2).
10. The juice is filtered by the second-carb filters.
11. If the factory is equipped with safety (security) filters, the juice is filtered again.

The amount of MOL entering the intermediate-liming tank (X) and mainliming tank (Y) can be calculated from the following equation (refer to Figure 3.10.10 for point numbers):

$$X = \frac{(\text{CaO})_3 - (\text{CaO})_1}{(\text{CaO})_4 - (\text{CaO})_1} \times 100 \qquad (3.10.4)$$

$$Y = 100 - X$$

DDS Purification System

DDS juice purification (see Figure 3.10.12) was developed by the Danish Sugar Company. It consists of preliming, cold mainliming, and first and second carbonation. The DDS system uses **thickening filters** instead of clarifier to separate the first-carb mud from the first-carb juice. The system works efficiently and has a lower lime requirement than other systems.

EXAMPLE 9

Given:

Amount of CaO in juice leaving the mixed-juice tank (point 1)	0.6% CaO on juice
Amount of CaO in juice leaving the intermediate tank (point 3)	1.0% CaO on juice
Amount of CaO in juice leaving the mainliming tank (point 4)	1.6% CaO on juice

Calculate:

Amount of MOL entering the intermediate tank (X) and the mainliming tank (Y) is

$$X = \frac{1.0 - 0.6}{1.6 - 0.6} \times 100 = 40\%$$

$$Y = 100 - 40 = 60\%$$

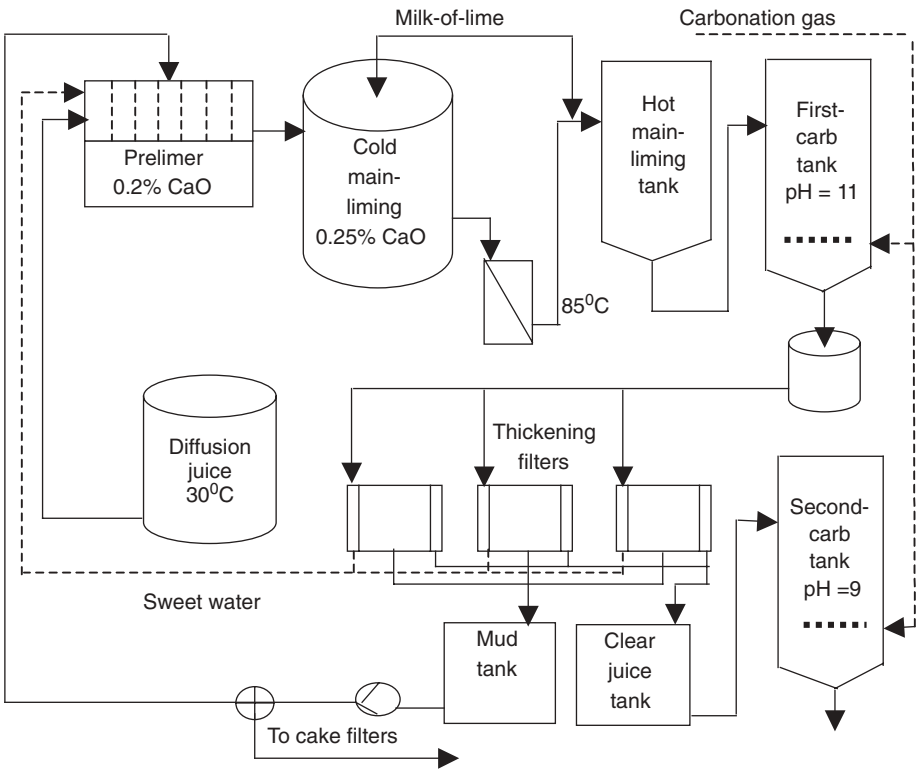


Figure 3.10.12 DDS juice-purification system

In the DDS system, the diffusion juice goes through the following steps:

1. The diffusion juice enters a horizontal prelimer.
2. Part of the first-carb mud (about 10%) returns to the second or third compartment of the prelimer. Partial return of the first-carb mud creates stable precipitates.
3. Prelimed juice from the prelimer is introduced to a cold mainliming with a long detention time (30 to 60 minutes), which produces a low-color juice.
4. The limed juice is heated.
5. The heated-limed juice goes for a 10-minute hot mainliming.
6. The mainlimed juice is carbonated in a single-pass carbonator. When processing damaged beets, a small amount of lime can be added to the first carbonation.
7. The juice is then clarified with a filter thickener, and the mud is separated on rotary-drum filters or filter presses.
8. The clear juice is second carbonated.
9. The juice is filtered by second-carb filters.

Dorr Purification System

Most beet-sugar factories in the United States use the Dorr system with some modifications. The original Dorr system has a Benning carbonator. The following general description of a Dorr system (see Figure 3.10.13) does not necessarily apply in all details to every modification.

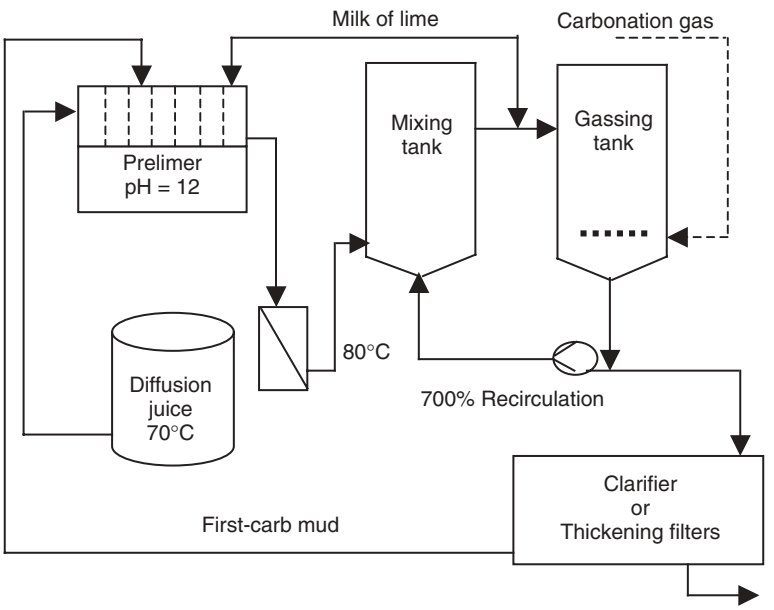


Figure 3.10.13 Dorr juice-purification system

In the Dorr system, the diffusion juice goes through the following steps:

1. Heated diffusion juice (usually to 70°C) enters a horizontal prelimer with seven sections (compartments). Lime and part of the first-carb mud are mixed with the juice in the prelimer to increase the juice pH to about 11.
2. The juice is heated to about 80°C.
3. The first-carb equipment has two tanks: *liming* and *gassing*. Prelimed juice enters the bottom of the liming tank, together with a large recirculation (up to 700%) from the gassing tank. The juice flows upward, leaves at the top of the tank, gets fresh lime, and enters the top of the gassing tank. In the gassing tank, the juice is mixed with gas and pumped by the recirculation pump back into the mixing tank. The larger part of the juice is pumped back to the mixing tank and the smaller part leaves the gassing tank as first-carb juice.
4. The juice is clarified with a clarifier or filter thickeners.
5. The clear juice is second carbonated.
6. The juice is filtered by second-carb filters.

Some factories still use the Dorr system with the original Benning carbonator (see Figure 3.10.14). The Benning carbonator is a single tank with two sections (mixing and gassing) that looks like a smaller tank in the middle of a larger tank. The mixing section is 1.5 m larger in diameter than the gassing section. For better mixing, the space between two sections is divided into several compartments by perforated partitions. In this system, MOL and gas are added directly to the carbonator. The diffusion juice flows into the recycled-juice pipe. The larger part (up to 700% on incoming juice) of the carbonated juice returns to the space jacket surrounding the carbonator. A small part of the carbonated juice (limed and gassed juice) leaves the lower part of the tank as first-carb juice.

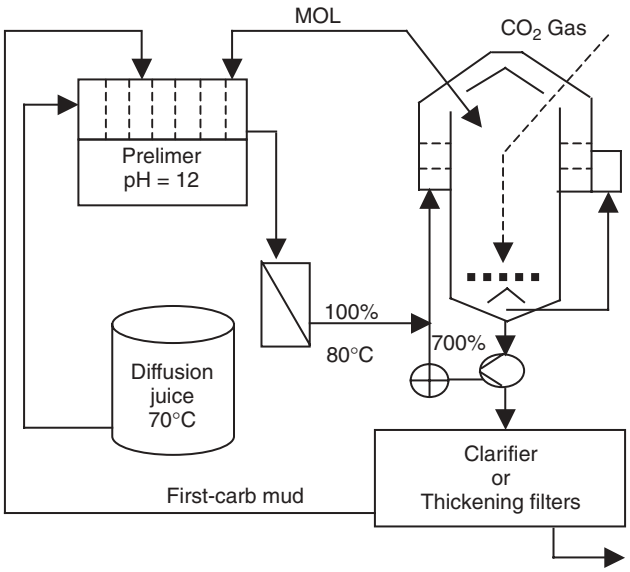


Figure 3.10.14 Dorr juice-purification system with Benning carbonator

ADDING CHEMICALS DURING PURIFICATION

Chemicals are added to the second-carb juice before filtration is practiced when the juice has

- A high pH drop during evaporation (more than 0.4)
- Low effective alkalinity (lower than 0.005% on juice)
- High hardness
- All of the above

The chemicals usually added during the juice-purification process are the following:

- Soda ash (Na_2CO_3)
- Caustic soda (NaOH)
- Magnesium oxide (MgO)

The addition of chemicals has the following advantages:

- Increases the pH of the juice
- Decreases the hardness of the juice
- Increases the natural and effective alkalinity of the juice

The reason for these points is that the chemicals decrease the limesalt solubility and replace Na (in the case of soda ash or caustic usage) for Ca, which lowers limesalts and increases alkalinity (the buffering capacity) and pH of the juice. In addition, sodium increases lime-reaction ability. The addition of **soda ash** (Na_2CO_3) is helpful, particularly when the beet quality is low, because it

- Increases juice pH
- Increases juice alkalinity
- Decreases juice hardness
- Decreases scaling in the evaporators

The nonscaling sodium in soda ash replaces scaling calcium and magnesium ions in a stoichiometric amount. In factories equipped with thin-juice softening, the hardness reduction helps the softening process (see Chapter 6).

$$Na_2CO_3 + Ca^{2+} \rightarrow CaCO_3\downarrow + 2Na$$

Note: The overdosing of soda ash has a major disadvantage (see also the following notes) with respect to sugar loss to molasses. It considerably increases melassigenic (molasses-producing) actions because Na is more melassigenic than all cations of the juice except potassium. The **melassigenic coefficient** (amount of sucrose carried into the molasses by each kg of nonsucrose) of soda ash is high (0.95 kg sucrose lost per kg sodium).

If the **natural alkalinity** of the juice is low (called low-buffered juice), a greater pH drop across the evaporators and sugar-end operations happen. Soda ash increases the alkalinity and pH of the juice by combining excess sodium with bicarbonate present in the juice:

$$Na_2CO_3 + CO_2 + H_2O \rightarrow 2NaHCO_3$$

The soda ash dose is calculated in Chapter 6. According to these calculations, the amount of soda ash required to bring the limesalt content down by 50% (e.g., from 200 to 100 mg/*DS*) is equal to about 0.5 kg (1.1 lb) per ton of beet processed.

Magnesium oxide (MgO), known as Magox, is also used as a hardness (limesalts) reducer and a pH increaser and stabilizer. It is added to the second-carb reaction tank at the amount of 0.06 kg/t beet (0.12 lb/t beet). The addition of MgO has the following benefits:

- Increases natural alkalinity and decreases pH drop of the juice during evaporation
- Decreases molasses purity (unlike soda ash) because magnesium ions are less melassigenic than sodium by about half
- Increases absorption action (it is a silica absorbent), reducing scale formation in evaporators

NOTES

- **The soda ash dose is important. At high dose, the sodium cations (Na^+) become, by far, the major (in terms of quantity) and the most melassigenic (in terms of quality) nonsucrose in the juice, which increases sugar loss to molasses. *This increases pH and viscosity excessively, causing operational difficulties in crystallization*.**
- **If the factory is equipped with molasses desugaring by chromatographic process (MDC process), which requires soft molasses (see Chapter 8), the process of thin-juice softening by ion-exchange resin (see Chapter 6) is required.**

PURIFICATION STARTUP PROCEDURE

The following **practical points** are helpful when starting the purification station at the beginning of the campaign:

- Check equipment (including the diffuser and all steps of purification) with hot water.
- Check gas and lime supplies.
- Drain all equipment except the diffuser.
- Start feeding the diffuser.
- Adjust the dry substance of the diffusion juice.
- Start adding lime. (For a dark-color juice, increase the lime, within reason. More lime and gas use is better than not enough, but in any event, do not use lime under 1.2% on juice.)
- Run the process with higher alkalinity during startup.
- Check the sedimentation rate of the carbonated juice. (Foamy, slow-settling juice with a small amount of mud indicates insufficient lime; the juice needs more lime or less gas.)

PURIFICATION OF DAMAGED BEETS

In the **diffusion section** (Station 7), you learned how to operate the diffusion station when processing damaged beets. Here, I explain how to run the purification station when the factory processes badly damaged beets, sometimes known as **Z-beets** (see Figure 3.7.11 in Section 7 of Chapter 3).

Many technological problems in the sugar factory are connected to the **colloidal substances**. Colloids are always in the diffusion juice. But when their presence reaches the extreme, we have to know how to fight them or, as some old sugar technologists tease, "close the factory and go home."

When beets are damaged, microorganisms, particularly the *Leuconostoc* group, consume the sugar and convert some of it to colloids (e.g., dextran and levan). At the same time, part of the sugar hydrolyzes to **invert sugar** and further to lactic and other acids. Normal purification can precipitate high amounts of acids but not enough colloids, causing operational difficulties. For example, when the dextran level gets high (above 100 ppm), the following problems occur:

- Filtration and settling difficulties because of high juice viscosity
- Production of thin juice with high hardness content
- Production of low-purity thin juice
- Plugging of the filter media

When Z-beets have to be processed, the following tips are helpful to better handle the situation:

- Switch from classical to defeco-carbonation purification if the factory has this choice. (Some factories with classical purification have equipped their first-carb tanks with lime-addition and juice-recirculation equipment. Therefore, if necessary, preliming and mainliming can be omitted, and the process changed to defeco-valuecarbonation.)
- Keep the **alkalinity** of the first-carb juice at a lower range (0.06 to 0.07) to improve the settling and filtration processes. Obviously, low alkalinity pushes the pH value down-ward.
- Skip mainliming when beets are badly damaged to prevent high alkalinity.
- Add lime (0.4 to 0.5% CaO on beet) during the second carbonation to increase adsorption.
- Use sulfitation in more stages than in the normal situation to improve juice color.
- Increase the amount of **settling aids** to improve the sedimentation rate.
- Adjust the retention times at different steps of the purification station.
- Increase the amount of **filter aids** to improve filtration rate.
- Add soda ash to the juice to decrease the hardness content.
- Add dextranase to the diffuser.

PRODUCT AND BY-PRODUCT OF THE PURIFICATION STATION

Thin juice is the product and **carbonation-lime residue** (lime residue or limecake) is the by-product of the purification station. Thin juice has the following properties:

- **Color:** Thin juice is **brown-yellow** (its darkness depends on the products of the decomposition of invert sugar formed during purification).
- **Dry substance:** Thin juice *DS* is about 0.5 units (average) lower than diffusion juice because of dilution and nonsucrose elimination during purification.
- **Purity:** Thin juice is about 3 units higher in purity than diffusion juice.
- **Hardness:** Thin juice hardness is 100 to 200 mg/100 *DS* at normal operation and when good beets are processed.

- **Colloid content:** Thin juice contains fewer colloids than diffusion juice because proteins, saponins, and pectins are mostly removed during purification.

In general, thin juice should be handled gently in the evaporation station. Rapid evaporation under vacuum is essential to achieve low-color thick juice with minimum sucrose inversion.

Carbonation-lime residue (limecake) is residual calcium carbonate from further concentration of carbonation mud in cake filters (filter presses or rotary drum filters). It is not environmentally friendly if it has to be accumulated in the lime pond of the factory rather than sold. Agricultural demand for lime residue depends on local soil needs. Some sugarbeet factories sell it as a by-product, while others have no market and must pay to landfill it.

The residue produced by **filter presses** (plate-and-frame filters) contains about 70% dry substance and is already in the form of cake and ready for sale. The residue produced by rotary-drum filters contains lower *DS* content (so it still looks like mud) and therefore should be kept in the factory lime pond to lose more water until it becomes wet cake and suitable for transport. After the lime in the pond has settled, the water is decanted from the top and sent for wastewater treatment.

In factories that use rotary-drum filters (produce lime residue with about 50% moisture content), the amount of carbonation lime (total amount of lime used and removed nonsugars during purification) is about 10% OB. In factories equipped with filter presses (produce lime residue with about 30% moisture content), the amount of lime residue produced is about 7% OB.

The approximate composition of carbonation-lime residue follows:

Carbonation-Lime Residue Composition (% on *DS*)

Calcium carbonate	80
Nitrogenous compounds	6
Minerals	6
Sucrose	0.5
Others	7.5
Moisture	30%
Bulk density	1100 kg/m^3 (70 lb/ft^3)

Because of the high content of calcium carbonate and other substances (e.g., nitrogen and minerals), carbonation lime residue is ideal for the following uses:

- Agricultural soil enhancer
- Soil pH adjuster

Carbonation lime not only limes the soil but also contains minerals (in addition to calcium) that give extra value to the soil. Carbonation lime has proved, invaluable in growing sugarbeet. Moreover, it is less expensive than other lime fertilizers.

MICROFILTRATION AND ULTRAFILTRATION OF THE JUICE

Microfiltration (MF) and **ultrafiltration** (UF) use semipermeable, porous membranes to separate some of the components of a solution. Both have been researched by the sugar industry to carry out some of the juice-purification functions. In the United States, the membrane

process had been under research on a pilot testing for a few years prior to 2005, with some funds provided by several sugar companies. Consequently, imbalanced cost/return and risk/return ratios, low practicality, and high complexity of the process have not convinced the executives of our industry to consider the full-scale installation of the membrane technology in the near future.

Lime has been used in classical juice-purification for more than two centuries. As was stated previously, although this process has several outstanding advantages, it produces carbonation-lime residue (CLR), which environmentally concerns the beet-sugar producers. This is so because if CLR is not sold it must be stored in the lime pond of the factory. The lime pond takes a lot of room and needs a great deal of cares. To cover the disadvantage of the classical-juice purification, several techniques have been investigated but none has proved to be as efficient as the classical method, so lime purification has remained the method of choice.

MF and UF were applied on a pilot scale to remove suspended solids and colloids from the diffusion juice (diffusion juice contains 0.5 to 1.5% suspended solids) and to reduce its turbidity. Typically, it could increase the purity of the juice by about 0.5 units. This equates to 5% nonsugar elimination (*NSE*), which is not great compared to conventional purification with a typical *NSE* of 15 to 30%. As a result, the membrane technology must be followed either by conventional purification process or by combination of purification and chromatographic process to considerably improve the purity of the juice. The membrane technology works on the **reverse osmosis** principle (discussed in Chapter 12). Membrane pore size in MF is usually 0.5 to 5 μm (micron) and in UF is 0.001 to 0.5 μm. UF membranes are often characterized by a **molecular weight cutoff** (MWC) based on measurements of fraction rejected (**retentate**) versus molecular weight. Molecules larger than cutoff size are rejected.

Membranes are made from porous ceramic or stainless steel, and are available in multiple-channel configuration. Each channel (Figure 3.10.15) is 5 to 25 mm in diameter. During the operation, the juice permeates through the membranes to form the **permeate** stream, which represents the product. The **retentate** can be used as an animal feed or recycled to the process.

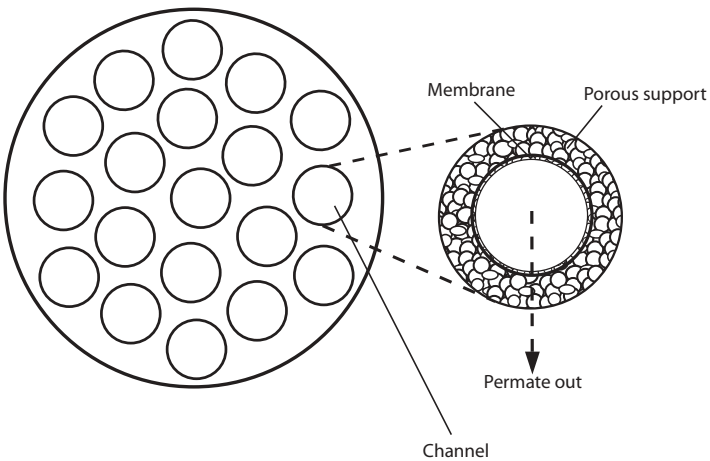


Figure 3.10.15 Cross sectional scheme of a membrane channel

The difficulties associated with the membrane technology are the following:

- Membrane fouling
- Low permeate flux
- Short membrane lifetime
- High cost of membrane filters
- High sugar loss to the retentate
- Low reliability of membrane functionality

During the membrane operation, a gradual decrease in the permeate **flux** (the rate of flow per unit area) causes membranes fouling. This is because of the change in the **osmotic pressure**, which has a major effect on the flux. The flux is improved by pretreatment of the diffusion juice before entering the membrane process. The main advantage of the membrane filtration of the diffusion juice is the reduction of lime use, which eases the environmental concern of beet-sugar producer. In view of all the pros and cons, including the membrane technology into the conventional juice purification can be a forward-thinking operation, but many problems have yet to be solved.

OPERATING PROBLEMS

A number of **problems** are encountered in the juice purification process. Most of the problems relate to the chemistry involved in purification.

Excessive Foaming during Preliming and Mainliming

In the preliming and mainliming operation, excessive foaming can occur. The following are possible causes of this problem:

- Not enough lime has been used
- Not enough defoaming agent has been added
- The retention time of the juice in the prelimer is too small
- The operation temperature of preliming and mainliming is high
- Beets grown under above-normal temperatures are being processed
- Damaged beets are being processed (there is a high amount of saponin in damaged beets)

Excessive Foaming during Carbonation

The following are possible causes of excessive foaming during carbonation:

- Rapid change in the flow rate of lime or carbonation gas
- Not enough defoaming agent has been added
- Insufficient CO_2 content in carbonation gas

Difficulties in Keeping Alkalinity at a Normal Range

Sometimes is hard to keep the alkalinity of first-carb juice at normal range (0.08 to 0.11). One or more of the following might be the cause of this problem:

- Unsteady juice flow rate
- Unsteady CO_2 gas flow rate
- Unsteady milk-of-lime flow rate
- Unsteady milk-of-lime concentration
- Incorrect temperature during operations
- Low- and high-quality beets are being processed together

Processing beets of different quality is usually observed toward the end of the campaign when healthy and damaged beets are processed. Because the amount of lime required depends on the quality of the sugarbeet, the difference in beet quality upsets the purification operation. This difficulty can be managed by closely watching the diffusion-juice purity and adjusting the lime addition accordingly (see the Lime Requirement section above).

Low Sedimentation Rate in the Clarifier

The optimum sedimentation rate for the best settling of a first-carb juice produced from normal quality beets is 4 to 6 cm/min (1.6 to 2.4 in./min). A sedimentation rate below 2 cm/min (0.5 in./min) is considered low. The following are the usual reasons for a low sedimentation rate:

- Damaged beets are being processed
- The operation temperature of the clarifier is low
- The first-carb juice is undercarbonated: juice is gassed inadequately, creating a high alkalinity (above 0.11) and a high pH (above 11.1)
- The lime use is high: the lime used for healthy beets should not be more than 2% on beet.
- The recirculation rate during the first carbonation is high
- The flocculent use in the clarifier is not sufficient (use of about 0.0008% OB flocculent is recommended)
- The clarifier capacity is insufficient: 60 to 90 m^2 (650 to 970 ft^2) settling area is required to handle the first-carb juice produced from 1000 tons beets per day

Low Gas Utilization in the Carbonation Process

Under normal conditions, the percentage of CO_2 utilization (gas absorption) ranges from 75 to 85%. There are several possible causes for low utilization of the carbonation gas:

- High juice temperature: CO_2 is less soluble in hotter solutions because high temperatures decrease the solubility of CO_2 in the juice, resulting in lower gas absorption by the juice.
- Low gas pressure: CO_2 is less soluble under lower pressure. A sufficient pressure differential (at least 1.0 psig which is equal to 1.07 atm) must exist for smooth gas flow.
- A low juice level in the carbonation tank decreases gas absorption.

First-Carb Juice Is Overcarbonated

If the first-carb juice is overcarbonated, it probably has been gassed too much. Overgassing causes the alkalinity of the first-carb juice to fall below normal (below 0.07 alkalinity and 10.7 pH) and creates other difficulties:

- Produces a juice that contains colloids, since at low alkalinity, some of the destabilized colloids return to their original forms
- Produces a high-color juice
- Produces a juice with a higher hardness content
- Produces a juice that contains **oxalic acid** ($H_2C_2O_4$) due to the precipitation of **calcium oxalate** (CaC_2O_4) scale in the evaporators and vacuum-pan crystallizers

A juice with low alkalinity provides a good settling and filtration rate. But in sugar operations, *low alkalinity is not recommended* because it creates low-quality juice. First-carb alkalinity must be maintained at optimum conditions to prevent creation of a bottleneck in the settling and filtration operations. The optimum alkalinity of first carbonation is between 0.08 and 0.11 (pH of 10.8 to 11.1).

First-Carb Juice Is Undercarbonated

If the first-carb juice is undercarbonated, it probably has not been gassed enough. Undergassing raises the alkalinity of the juice to above normal (above 0.11 alkalinity and 11.1 pH) and creates other difficulties:

- Produces a juice with a high amount of foam
- Produces a juice with a low sedimentation and filtration rate
- Creates high sugar losses because the juice was not gassed enough to decompose all the *calcium saccharate*, which ends up in the carbonation-lime residue

Difficulties in Maintaining Alkalinity

Some further difficulties in maintaining alkalinity may be due to one of the following:

- Shortage of carbonation gas: This problem happens when low-quality beets are processed (low-quality beets need more lime and gas to be processed) or where there is insufficient clarifier capacity, or both. This problem can be solved by running carbonation at a higher temperature, which increases the solubility of CO_2 in the juice and thus gas absorption by the juice.
- Low gas pressure
- Plugged gas distributor
- High juice concentration
- Plugged in-line pH electrode
- Erroneous laboratory analyses
- The first-carb recirculation rate is not maintained properly

High Limesalts in Carbonated Juice

Primary causes of high limesalts are the following:

- Insufficient lime addition
- Improper carbonation
- Improper filtration

CHAPTER
3

SECTION 11

SEDIMENTATION AND FILTRATION

SUBSECTIONS

- Carbonation-Mud Separation
- Carbonation-Mud Thickening
- Types of Filters
- Filter Cloths
- Filter Aids and Settling Aids
- Operating Problems

ABOUT THIS SECTION **This section** talks about **Station 11** (Sedimentation and Filtration) of sugarbeet processing. Station 11 (see Figures 3.11.1 and 3.11.2) finishes the job of Station 10 (Juice Purification). Here, solid particles formed during purification are separated thoroughly to produce clear thin juice. If sedimentation (settling) and filtration are not performed properly, the juice-purification station is affected, which may slow or shut down other stations as well.

In the preceding section, you learned how the diffusion juice is limed, that is, milk of lime [$Ca(OH)_2$] is added. You also learned how the limed juice is carbonated, that is, carbonation gas (CO_2) added. In addition, you learned that the high amount of lime used during purification process (1.0 to 2.5% CaO on beet) converts again to calcium carbonate ($CaCO_3$) precipitate (↓) when the juice is carbonated ($CaO + CO_2 \rightarrow CaCO_3\downarrow$).

You are now ready to learn how the precipitates are sedimented and filtered to produce a clear juice that can be processed in the evaporators (Station 13).

Beet-Sugar Handbook, by Mosen Asadi
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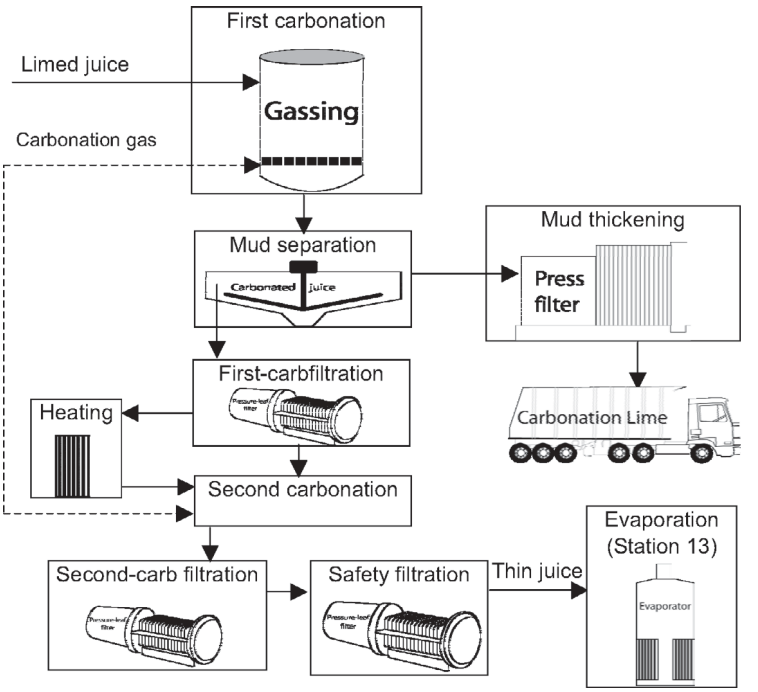


Figure 3.11.1 Flow diagram of sedimentation and filtration (Station 11)



Figure 3.11.2 Pressure-leaf filters (U.S. filters), the most-used filters in the sugar industry. [In the photo is Hans Schwieren (1935–2005), a legendary sugar-engineer with over half a century's service to the German and U.S. beet-sugar industries.]

Sedimentation (settling) is a rough way of separating solid particles from a liquid by gravity. The heavier of two phases (the solid phase) settles to the bottom while the lighter phase (the liquid phase) stays at the top. The equipment used in the settling process is called a **clarifier** (decanter).

As was stated in the previous sections, after the first-carbonation process, the juice becomes a slurry with suspended solids, called the **first-carbonation slurry** (later, **first-carb slurry**). The suspended solids in the slurry are visible to the naked eye and consist of precipitated calcium carbonate (**PCC**) and nonsugars released by the liming process.

Before the 1970s, most sugar factories used **plate-and-frame presses** (today's automated form of these presses is called membrane filter presses) in the direct filtration of the first-carb slurry. Because of the high solid content in the slurry, direct filtration was not entirely satisfactory, so sugar factories started to use a two-step process:

- **Carbonation-mud separation:** In this step, the solids in the slurry are separated in the clarifier or thickening filters to produce **carbonation mud**.
- **Carbonation mud thickening:** In this step, the carbonation mud is thickened (dewatered) with **cake filters** (filter presses or rotary-drum filters). The filter cake residue is called **carbonation-lime residue** (limecake), which is trucked to the factory's lime pond to be sold to customers as a byproduct (see the Note below).

Filtration is the process of separating solid particles from a liquid by the force of the pressure difference on both sides of the **filter medium** (a porous material). The **filter residue** is the solid that remains on the filter medium, and **filtrate** is the liquid that passes through the filter medium and is collected during filtration. The filtration process is used during juice purification two to three times. The amount of precipitate produced during the first carbonation is much higher than during the second carbonation. After the first and second carbonations, the precipitated calcium carbonate (**PCC**) and nonsucrose substances (nonsugars) are filtered to produce clear juice, known as **thin juice**. In some factories, the filtered second-carb juice is filtered again by safety filters.

NOTES

- **The carbonation-lime residue produced by membrane filter presses (automated type of plate-and-frame filters) contains about 70% solid content, and that produced by rotary-drum filters has about 50% solid content. Usually, the lime residue produced by rotary filters is kept in the lime pond to lose more moisture.**
- **Carbonation-lime residue is a by-product of the factory and is usually sold for a variety of uses but mainly as a soil pH enhancer and a lime fertilizer.**

CARBONATION-MUD SEPARATION

In the **mud-separation step**, the solids present in the first-carb slurry are separated to produce a concentrated solid known as **carbonation mud**. The carbonation mud is separated from the first-carb juice for the following reasons:

- **To help the filtration process:** The juice contains so many solids that numerous filters would be needed if the mud were not separated.

- **To lower the cost of filtration:** The cost of the filtration is lower than if the first-carb juice were filtered without the mud-separation process.
- **To help the carbonation-mud recycling:** Separation of the first-carb solids produces a thicker solid that is more suitable for recycling to the prelimer (see Section 10 of Chapter 5).
- **To help the carbonation-mud caking:** The thickening process helps the caking process in the cake filters (rotary-drum filters or filter presses).

Depending on the type of purification systems used by the factory, one of the following is used to separate mud from the first-carb juice:

- Clarifier
- Thickening filter

Clarifiers are used in almost all purification systems with **progressive liming** (e.g., the BMA and Dorr systems), but they may produce an unclear overflow when used with **full-liming** systems. Therefore, factories that use full liming usually use thickening filters.

Clarifiers

Clarifiers (settlers or thickeners) work based on **gravity sedimentation**. The sedimentation (settling) process is also called **clarification** because it produces clarified liquid.

The majority of sugar factories use continuous clarifiers to separate solids from the first-carb slurry. The overflow from the clarifier is called **clarified juice**, and the underflow is **carbonation mud**. Because of settling, about 75% of the first-carb slurry is converted into **clarified juice** (clarifier's overflow), which is nearly free of suspended solids, and 25% becomes mud (clarifier's underflow). The volume ratio of the mud to the slurry is called **thickening ratio**.

In most purification systems, 50 to 60% of the mud returns to the prelimer, which is known as **carbonation-mud recycling** (see Station 10). The rest of the mud is filtered by cake filters (filter presses or rotary vacuum filters) for further concentration to produce limecake (carbonation-lime residue). The overflow is pumped either directly to the second carbonation or to filtration and then to the second carbonation.

Settling (sedimentation) **rate** is the height of the settled particles per unit time. A low settling rate (below 2 cm/min or 0.8 in./min) in the clarifiers from healthy beets is generally an indication of an improper purification process. The best indicators of proper clarification are the following:

- Reasonable settling rate (4 to 6 cm/min or 1.6 to 2.4 in./min)
- Reasonable concentration of the mud (about 20° Baume)
- Reasonable thickening ratio (0.2 to 0.4)
- Reasonable clarity in juice overflow

Normally, the mud concentration can be maintained between 20° to 22° Baume (36 to 40% *DS*) for optimum performance. When low-quality beets are processed or a clarifier with insufficient capacity is used, lower values can be accepted.

Let us see what happens during the settling of suspended solids in a clarifier. At first, the solids are uniformly distributed in the juice to form one zone (**A** zone), as shown in case 1 of Figure 3.11.3. As settling proceeds, different zones are formed. After a short time, the solids

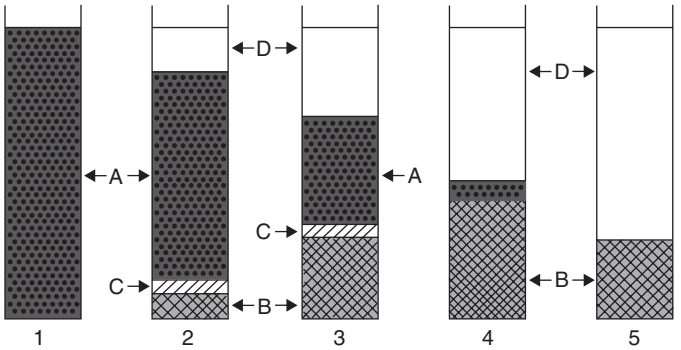


Figure 3.11.3 Sedimentation

gradually start to settle to the bottom and form a zone of settled solids (bottom zone, or **B** zone), and a zone of clear juice (top zone, or **D** zone). Above zone **B** is a zone of unclear juice (middle zone, or **C** zone), in which the solid concentration is not similar to the top or bottom zone and still contains some solids. As settling continues, the top and bottom zones increase in depth, and the middle zone decreases. Eventually, the middle zone disappears, and almost all the solids are now in the bottom zone in a concentrated form known as **mud**. Note: The boundaries of the middle zone (**C**) with its upper and lower zones may not be distinct, but the boundary between the bottom (**B**) and middle (**C**) zones is sharp.

The settling rate in the clarifier is affected by a number of factors:

- **Alkalinity:** Although the settling rate is improved by low alkalinity, running first carbonation at below-normal alkalinity is not recommended because it produces a juice with high color and **hardness** (limesalts), which is not desired. Therefore, a compromise must be made by choosing a practical alkalinity (0.08 to 0.10) without creating difficulties for the settling and filtration operations. Among other problems, running first carbonation at low alkalinity (below 0.07) redissolves the precipitate, which lowers the juice purity (increases sugar losses in carbonation lime residue). By regularly performing settling tests (explained later in this section), you can gain valuable guidance for clarifier operation.
- **Amount of lime and purification method:** Normal lime use, based on the purity of the juice, and **defeco-carbonation purification** (progressive preliming and simultaneous liming and gassing during first carbonation) are recommended. Defeco purification creates more compact particles than classical purification. Compact particles have larger density, so they have a faster settling rate.
- **Temperature:** Settling becomes faster with increasing temperatures of the clarifier's feed, up to a certain point. The practical temperature is between 80 and 85°C.
- **Settling aids** (flocculent)**:** Flocculent (coagulant) is used to get particles closer to each other and help them settle faster in the clarifier. Generally, settling aids do not necessarily improve juice clarity but improve the settling operation by increasing the settling rate. Obviously, a faster settling rate increases the capacity of the clarifier. Therefore, use of about 0.0008% OB settling aid in the clarifiers is recommended.
- **Clarifier capacity:** A clarifier with a settling area of 60 to 90 m^2 (650 to 970 ft^2) is required to handle the first-carb juice produced from 1000 tons of beets per day. Such a settling area can even handle a low settling rate of 1.2 cm/min (2 m/h). However, undamaged beets with

good purification operations produce juice with a settling rate of 4 to 6 cm/min (7 to 10 m/h or 1.6 to 2.4 in./min).

The volume of clear juice produced by a clarifier depends primarily on the area available for settling, and on an industrial scale it is almost independent of the liquid's **depth**. Therefore, a higher capacity per unit of floor area can be obtained by using multiple-compartment clarifiers, which have a lower depth compared to the single-compartment clarifier. The compartments of a multiple-compartment clarifier are stacked one above the other. To determine the **settling rate**, the purification operator performs the following simple test:

1. Fills a 1000-mL graduated cylinder, marked in cm (or in.) increments, with the first-carb juice.
2. Starts timing the settling rate as soon as the sample shows the first sign of separation (a very thin layer of clear juice above the solids starts to occur).
3. After exactly one minute, reads the level of the settled solids (the boundary between clear juice and solids). The settling rate is the height of the settled solid in one minute.

With normal operations and when processing good-quality beets, a settling rate of 4 to 6 cm/min (1.6 to 2.4 in./min) is expected.

To control the operation of the clarifier, you can calculate the amount of the mud (X) and clear juice (Y). For this calculation, you use the density of the first-carb juice entering the clarifier (d_1), the density of clear juice (d_2), and the density of the mud (d_3):

$$X = \frac{d_3(d_1 - d_2)}{d_1(d_3 - d_2)} \times 100$$

$$Y = 100 - X$$

EXAMPLE 1

Given:

Density of first-carb juice (d_1)	1090 kg/m^3
Density of clear juice (d_2)	1050 kg/m^3
Density of mud (d_3)	1195 kg/m^3

Calculate:

% Mud produced in the clarifier (X)

% Clear juice produced (Y)

$$X = \frac{d_3(d_1 - d_2)}{d_1(d_3 - d_2)} \times 100 = \frac{1195(1090 - 1050)}{1090(1195 - 1050)} \times 100 = 30.2\%$$

$$Y = 100 - X = 100 - 30.2 = 69.8\%$$

Sugar factories use different types of continuous clarifiers for the rough separation of solids from the first-carb juice. Four types of clarifiers are the most common in sugar factories:

- Dorr clarifier
- Dorr-ATV clarifier

- BMA clarifier
- Enviro-clear clarifier

The **Dorr clarifier** (Dorr multi-feed clarifier) is a vertical tank, typically 8 to 12 m (24 to 36 ft) high and 5 to 6 m (15 to 18 ft) in diameter. It has an inverted-cone bottom and a covered top. The Dorr clarifier is a multiple-compartment unit (a four-compartment clarifier is seen in Figure 3.11.4). In multiple-compartment clarifiers, the settling compartments are built one above the other to obtain a higher capacity per unit of floor area. Compartments are separated from each other by inclined trays. Each tray contains a slow-moving **rake arm** to sweep the mud toward the central opening. A central shaft drives the rakes.

Hot first-carb slurry continuously enters the first compartment (first from the top), which degasses the juice and separates the foam from it. The feed is then passed into the center cylinder, and from there, into each of the lower compartments. In each compartment, the precipitate settles at the bottom of the tray. Settled mud from each compartment is swept by rake arms to the central opening and then into the mud tank, installed below the clarifier. From here, the mud is pumped by means of a diaphragm-type suction pump.

The clear juice flows slowly toward the header, which is located at the highest point. The header from each compartment is connected to a pipe that ends in the overflow tank, which collects the clear juice. The retention time is 15 to 45 minutes, and the required capacity is 0.5 to 1.0 m^3 per ton of beet per hour.

The **Dorr-ATV clarifier** (see Figure 3.11.5) might be considered four separate clarifiers, stacked on top of each other, that receive feed from the same feed tank. Feed is split equally to each section through a centrally located feed pipe. The settled mud is pumped out from each section by individual pumps.

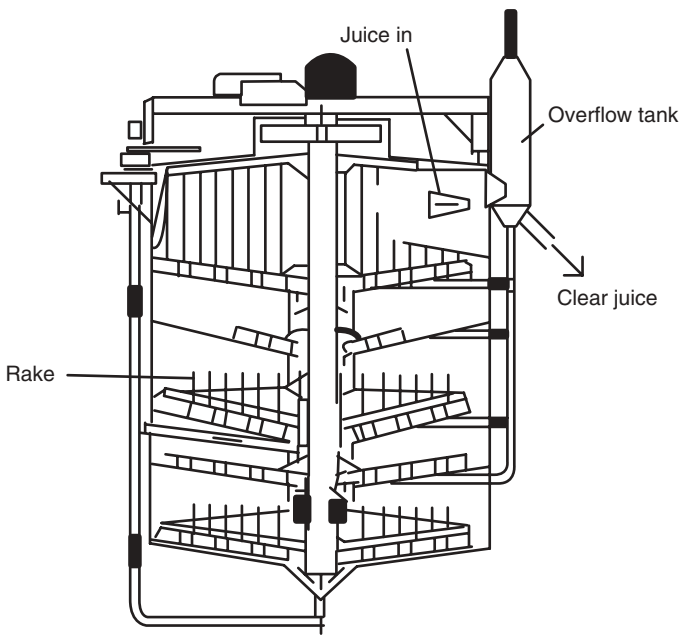


Figure 3.11.4 Dorr clarifier

The **BMA clarifier** is similar to the Dorr clarifier, but the compartments are not interconnected by a central opening. The hot first-carb juice feeds through a splitter into each compartment, gets more concentrated, and flows through the bottom of the inner chamber into the outer chamber for further settling. As the slurry goes to the bottom of the compartment, the clear juice moves out of the inner chamber. The mud then moves toward the central discharge by means of sweeps and is discharged by gravity. The gravity discharge avoids breaking the particles.

The **Enviro-clear clarifier** (see Figure 3.11.6) consists of a tank with slow-moving arms driven from a central shaft. The feed flows from an inclined channel into the center of the clarifier. As settling proceeds, the solids move downward by gravity, and then arms move the settled solids (mud) gently toward the center of the clarifier. In the center is an opening for the slurry discharge. Clear juice moves toward the outer part of the clarifier and spills into a container for collection.

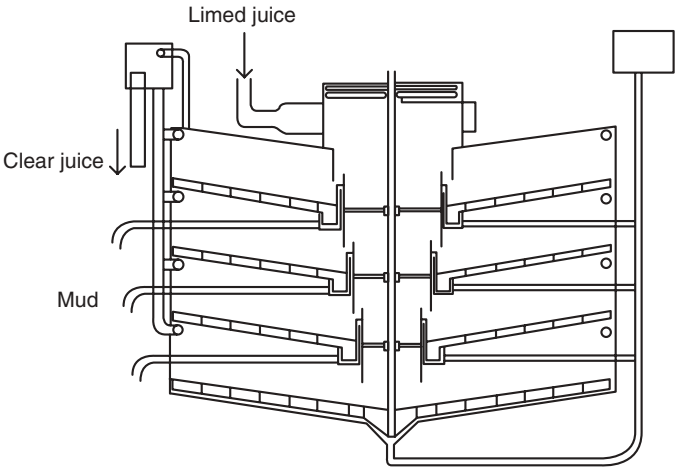


Figure 3.11.5 Dorr-ATV clarifier

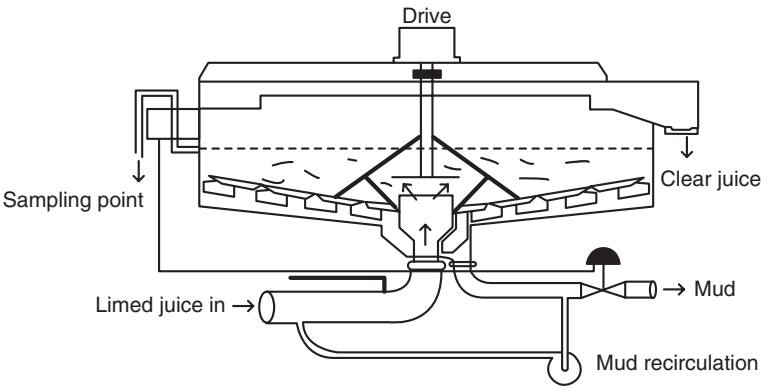


Figure 3.11.6 Enviro-clear clarifier

Thickening Filters

Some factories use thickening filters instead of clarifiers to produce carbonation mud. Kelly filter is the oldest type of thickening filters. The newer types, such as the Genter filters, operate by vacuum, and others, such as BMA, Grand Pont, and Putsch work with pressure. Some filter manufacturer offer thickening filters with stainless steel or ceramic candles, covered with filter cloth. In general, approximately 50 m^2 (177 ft^2) filter area is required to process juice from 1000 t beets per day.

Two types of thickening filters are generally used in sugar factories:

- Tube-thickening filter
- Bag-thickening filter

Grant Pont (see Figure 3.11.7) and Putsch (see Figure 3.11.8) filters are tube-thickening types. The operation of these filters differs, but the basic process is about the same. The limed juice enters the filtering tubes, and passes through the filter cloth, on which the solids deposit. When the mud builds up, it is released to the bottom of the cone and discharged out the filter.

NOTES

- **Clarifiers concentrate the slurry three to five times, and the thickening filters, five to seven times.**
- **Clarifiers are less expensive to operate than thickening filters.**

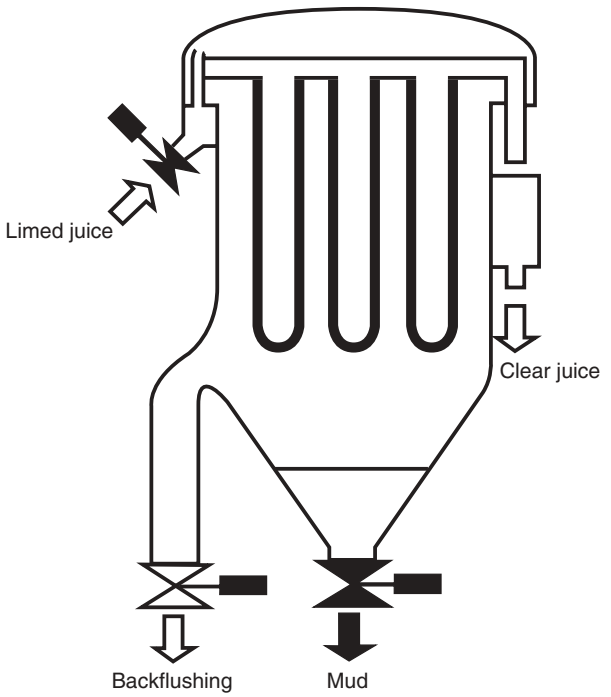


Figure 3.11.7 Grant Point thickening filter

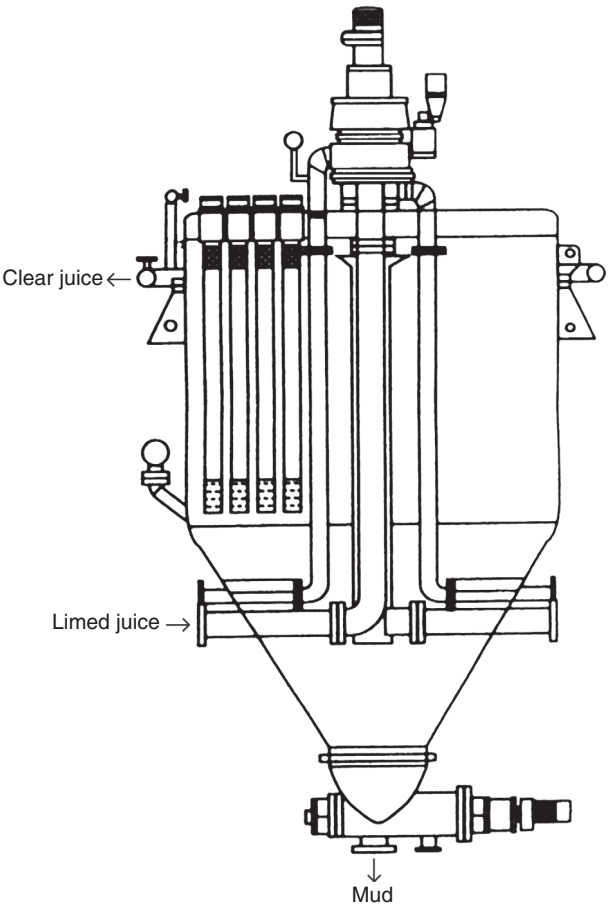


Figure 3.11.8 Putsch thickening filter

CARBONATION-MUD THICKENING

In the mud-thickening step, the **carbonation mud** (simply, mud) is further thickened in cake filters. The **cake filters** (filter presses or rotary-drum filter) are used to further concentrate the carbonation mud to produce the filter cake, which is known as **carbonation-lime residue** (limecake). The mud is thickened for the following reasons:

- To produce product suitable for transport
- To remove sugar left in the mud

Depending on the factory, one of the following cake filters is used to thicken carbonation mud:

- Filter presses
- Rotary-drum filters

Filter Presses

The oldest type of **filter press** is the **plate-and-frame press**. Plate-and-frame presses operate discontinuously and use pressure to filter and press the mud to produce cake. At the start of filtration in a plate-and-frame press, some solid particles begin to deposit on the filter medium to form a layer of thin cake. After this brief initial period, the cake does the filtration, not the filter medium. When a certain amount of cake is built up, the cake is washed to remove the remaining sugar. The filter cake is called **carbonation-lime residue** (limecake), which has a *DS* content of about 70%.

A plate-and-frame press consists of several sets of two square plates (one for juice and the other for water inlet) and a frame. The face of the plates is covered with filter cloth. The arrangement of plates and frame starts with a solid head and continues in the following way: juice plate, frame, water plate, frame, juice plate, frame, water plate, and so on. The sets sit vertically in a metal rack, and a screw squeezes them together. Each cycle consists of the following steps:

1. **Filtration:** Feed is pumped into one end of the assembly and passes through an inlet channel, which runs through the entire assembly. The channel feeds the frames, where the solids gradually are collected. The juice passes the filter cloth and the plates, enters the juice-outlet channel, and goes out from the discharge end. Filtration continues until juice no longer flows out the discharge end. When the frames are full of solid (it usually takes one hour), the press is said to be **jammed**.
2. **Cake washing:** The inlet for feed is closed and wash water (about 60°C) enters to desweeten the cake (removing sugar leftover from the cake) for about 15 minutes. The sweet water (it has about 70% purity) is pumped to the slaker to produce MOL.
3. **Cake drying:** Low-pressure steam or compressed air is blown through the cake to reduce the water content of the cake.
4. **Cake emptying:** The assembly is opened manually to drop the cake from the plates. Normally, the cake easily drops, but processing of low-quality first-carb juice can cause the cake to stick to the filter cloth. In these situations, the filter cake is removed by a scrapping blade.
5. **Cleaning:** The plates, frames, and filter cloths are cleaned for next cycle.

The cake from filter presses, called carbonation-lime residue, has about 60% *DS* and 0.6% sugar. The amount of CaO in the produced carbonation-lime residue is 7 to 10% OB, depending on the amount of lime used during purification. Plate-and-frame filters have the following disadvantages:

- High labor requirement
- Low *DS* content
- Low capacity

Membrane filter presses (or just filter presses) are the new generation (modified and automated) of plate-and-frame filters, designed in the 1970s to operate continuously and overcome the disadvantages of the plate-and-frame filters. A medium-size filter press can have 100 sets (50 membrane plates and 50 chamber plates). Plates are covered with polypropylene filter cloth. Figure 3.11.9 shows a typical filter press, which operates automatically with little labor and can be directed from the control room of the factory.

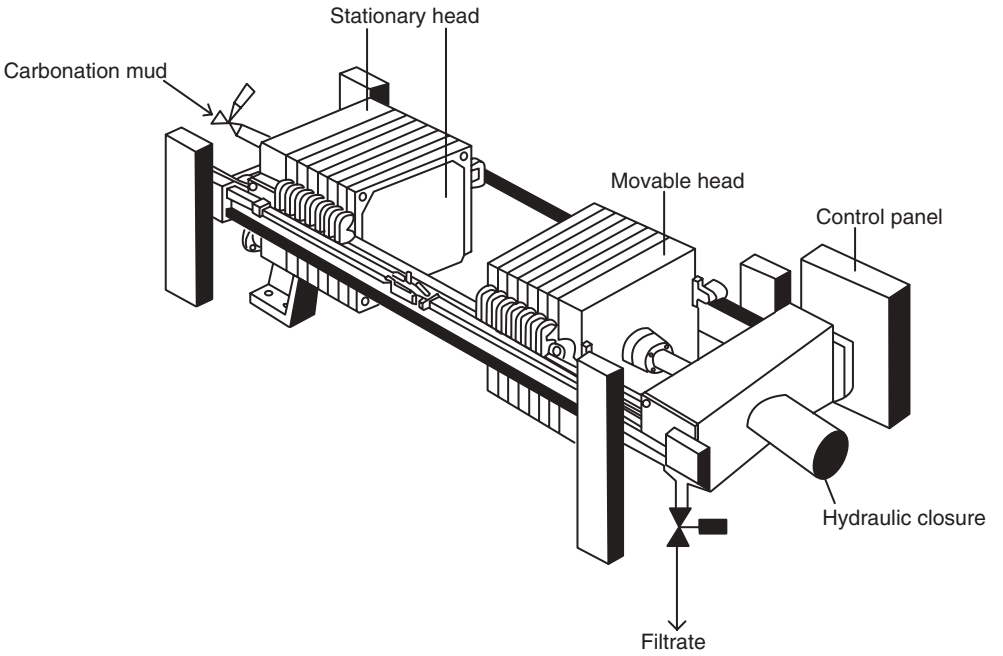


Figure 3.11.9 A typical filter press

Unlike the old filter presses, the sets of plates and frames in the membrane presses are squeezed by a hydraulic ram with a pressure of about 6 bar (88 psi). Like the old filter presses, the membrane presses work discontinuously, and each cycle usually consists of more steps than the original plate-and-frame presses. The steps in all types of membrane filter presses are not the same, but each cycle, which takes about 20 minutes, typically consists of the following steps:

1. Filtering
2. Light squeezing
3. Washing
4. Hard squeezing
5. Core blowing
6. Cake blowing

A filter press with 100 m^2 filter area can handle 2000 to 3000 t of beet processing a day. A few types of filter presses are available. Hoesch offer an antomatic filter press with a horizontal arrangement of plates. When the plates open, an endless traveling cloth removes the cake. In Putsch filter press (see Figure 7.4 in Chapter 7), the cake is discharaged by a mechanical cake-removal system.

The plates and frames in most filter presses are made from polypropylene. The face of the plate is covered with the filter cloth. The operations are carried out automatically, as follows:

1. The feed, from clarifiers or thickening filters, enters at the end of the assembly.
2. The feed passes through a channel running lengthwise through one corner of the assembly.
3. Auxiliary channels carry the feed from the main inlet channel into each frame.
4. The solids are deposited on the face of the plates in a form of cake.
5. The filtrate passes through the cloth, down into the plate faces, and out the press.
6. With increasing thickness of the cake, the pressure rises and the flow rate decreases. Filtration continues until the filtration pressure rises to a maximum set point (0.8 to 1.0 MPa). At this point, the typical thickness of the cake is about 4 cm.
7. Wash water is then admitted to desweeten the cake.
8. Steam is applied to displace the residual juice.
9. The press is opened and the filter cake (carbonation-lime residue) is dropped to a belt conveyor, which discharges the cake to a hopper, installed outside the main building.
10. Filter cloths are washed, preparing the filter press for the next cycle.
11. Lime residue is trucked to the lime pond of the factory or delivered to customers.

Rotary-Drum Filters

Rotary-drum filters (rotary-vacuum filters) work continuously and use vacuum to filter the mud. The cake on the drum is then sweetened with water and pumped to the lime pond. At this point, its dry substance (*DS*) content is about 50%. After the lime in the pond has settled, the water is decanted from the top and sent for wastewater treatment. The disadvantage of these filters, compared with filter presses, is that they cannot produce cake with low moisture content.

A rotary-drum filter, as shown in Figure 3.11.10, consists of a slotted drum (a round metal sheet with small holes) 2 to 3 m (6 to 10 ft) in diameter, a mud channel, a blade for removing the cake from the filter cloth, a vacuum pump, and connecting pipes. The filter cloth covers the face of the slotted drum (outer drum). Under the slotted drum is a smaller drum

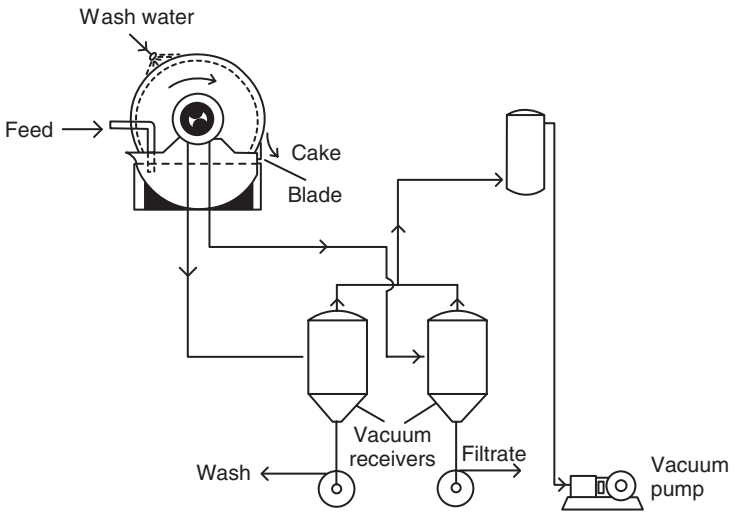


Figure 3.11.10 Rotary-drum filter operation

with a solid face. The slotted drum turns at 0.1 to 2 RPM in an agitated tank. Between the two drums are partitions dividing the space into two separate compartments. When the drum rotates, vacuum and air are alternately applied to each compartment. The operations in rotary filters are carried out, as follows:

1. The mud, from clarifiers or thickening filters, is pumped into the filter tank with a slow agitator to prevent settling.
2. From here, the mud is fed into the filter.
3. When the drum is in the mud, vacuum is applied, resulting in filtration and cake buildup.
4. When the drum leaves the mud and enters the washing part, again a vacuum is applied (from a different source) to suck wash the liquid from the surface.
5. After the cake has been dried, the vacuum is cut off; a little air is applied to crack the cake away from the filter cloth; and the cake is removed by a scrapping blade.
6. Once the cake is discharged, the drum enters the liquid again, and a new cycle starts.

Putsch and BMA produce rotary-drum filters with one compartment. The vacuum is applied to the entire inner surface of the filter cloth. The cake is discharged by airflow through the cloth. The amount of drum that is submerged is different from that of multi-compartment filters. Rotary-drum filters are less expensive than multiple-compartments filters, and the cloth can be changed more easily.

NOTES

- **Filter presses cost more to install than rotary-drum filters. However, modern factories prefer membrane filter presses over rotary-drum filters for the following reasons:**
 - **Sugar loss: Sugar loss to cake produced by filter presses accounts for about 0.7% on sugar entering the factory. With vacuum filters, the losses are 0.9 to 1.4%. Less sugar is left in the cake produced with filter presses because of better desweetening and better pressing mechanisms.**
 - **High dry substance content: Filter presses produce cake with about 70% *DS*, but rotary-drum filters produce cake with about 50% *DS*. Therefore, while in the lime pond, the product of drum filters need decanting to reduce its moisture content. This costs extra expenses.**
 - **Quality of filtrate: Filter presses can produce clear juice, but filtrate from rotary-drum filters still contains some suspended solids.**

TYPES OF FILTERS

In addition to rotary-drum filters, explained in previous section, the sugar industry uses some other types of filters, such as the following:

- Pressure-leaf (U.S.) filters
- Centrifugal filters
- Screen (Sibomat) filters
- Bag-pressure filters

Pressure-Leaf Filters

Pressure-leaf filters (U.S. filters or industrial filters) are the most common filters in the sugar industry. These filters operate continuously and automatically under high pressure in a closed system with mechanical cake discharge. The leaves of pressure filters are covered with filter cloth, and diatomaceous earth or perlite is used for precoating and body feeding as a filter aid. Pressure-leaf filters are cleaned internally, called filter sluicing, by nozzles mounted on movable arms.

Pressure-leaf filters have the most application in sugar factories for fine filtration, in which the amount of solid is low, creating relatively long cycles. The pressure-leaf filters are used in sugar factories for filtration of the following:

- First-carb clear juice
- Second-carb juice
- Second-carb juice safety filtration
- Thick juice
- Standard liquor
- Extract from molasses-desugaring operations

Pressure-leaf filters have a good **filtration rate** (the volume of the liquid to be filtered per surface area of filter medium per unit time). Pressure filters are made in a variety of designs. Most suppliers offer both horizontal and vertical designs. In the horizontal design, shown in Figure 3.11.11, leaves are held vertically on a retractable rack. (Note: The unit shown in the figure is open for discharge; during operation, the leaves are inside the closed tank.) In the vertical design, shown in Figure 3.11.12, the leaves are held horizontally. In both designs, the leaves are held together in a stack or cartridge. The steps in pressure-leaf filters are performed automatically as follows:

1. Feed is pumped through the side of the tank and passes the leaves, on which the solids deposit.
2. As the deposit forms, it is released from the filter cloth and allowed to remain on the bottom of the tank to settle further.
3. Thickened mud discharged from the filter is usually pumped to the mainliming.

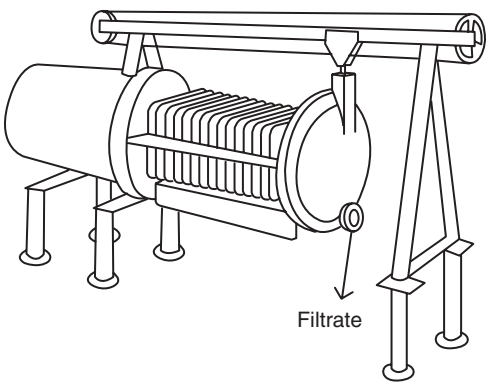


Figure 3.11.11 Horizontal pressure-leaf filter

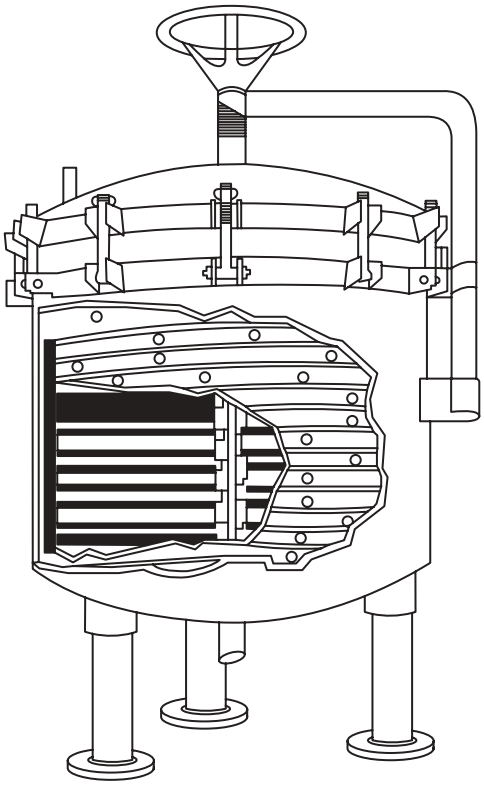


Figure 3.11.12 Vertical pressure-leaf filter

4. The filtrate passes through the leaves into a discharge manifold.
5. The feed stops and the cleaning cycle starts, with nozzles spraying wash water or clear juice (to avoid dilution).

Pressure-leaf filters are used for filtration with and without **precoating**. In the precoating operation, a layer of filter aid (e.g., diatomaceous earth), at a dosage of 0.05 to 0.2% on solids of the juice, is first deposited on the filter cloth. Feed is then pumped through the layer of filter aid, depositing a thin layer of solids. The filtrate may first come through cloudy and then improve to clear. Cloudy filtrate is returned to the feed tank for refiltration.

It is important to keep the nozzles from clogging. During precoating, some fine particles may get to the nozzle openings and clog them, so the nozzles should be monitored.

NOTE

Some factories use pressure-leaf filters in safety filtration (also called security filtration) after the second-carb filtration. In these factories, the filtered second-carb juice is filtered once more to ensure that no solid particles are left in the juice. In some modern factories, safety filtration is also provided after the sulfur dioxide (SO_2) treatment.

Centrifugal Filters

Centrifugal filters are used for filtration of high-concentrated liquids that can form a cake on the screen basket of the filter. Two types of centrifugal filters are used:

- **Disk centrifugal filters:** They are used to filter liquids with small amounts of fine-particle solids, such as thin and thick juice and standard liquor. Some sugar factories use these filters for safety filtration.
- **Decanter centrifugal filters:** They are used to filter liquids with large amounts of large-particle solids, such as slurry type liquids. Some sugar factories, such as the Mendota factory in California, use these filters to thicken the cake after rotary drum filters.

In general, centrifugal filters are similar to centrifuges and operate like them as well. These centrifuges consist of a perforated basket 0.5 to 1.0 m (20 to 40 in.) in diameter that rotates at approximately 5000 RPM. The basket looks like a funnel and is covered with a metal screen.

Batch centrifugal filters operate as follows:

1. **Feeding:** Feed is fed into the rotating basket.
2. **Filtering:** Pressure resulting from the centrifugal action pushes the liquor through the screen, leaving the solids behind.
3. **Rinsing:** The solids are rinsed by spraying hot water at appropriate intervals for a controlled length of time.
4. **Unloading:** The basket speed is slowed so that a heavy knife, which moves periodically, can scrape the solids.

The entire operation of the centrifugal filter is controlled by cycle timers and solenoid valves. The cake produced by a centrifugal filter is much drier than that of other filters.

Screen Filters

Screen filters (e.g., sibomat type from Putsch) are used in some factories with pressure-leaf filters for standard-liquor safety filtration. Unlike pressure-leaf filters, screen-type filters do not need diatomaceous earth as a filter aid because of their fine screen element (about 50 microns). This feature reduces the consumption of diatomaceous earth by a considerable amount. However, complete elimination of pressure-leaf filtration is not recommended.

Bag-Pressure Filters

Bag-pressure filters (see Figure 3.11.13) are used as safety filtration to filter the sediment from the juice. Factories equipped with molasses desugaring by chromatographic process (MDC process) use bag-pressure filters, which have extremely fine pores for safety filtration of feed (diluted molasses) to the separator.

FILTER CLOTHS

The filter cloth must meet general requirements for any type of filter and particular requirements for individual filters. In general, the filter cloth must have the following properties:



Figure 3.11.13 Bag-pressure filters for fine filtration

- Retain solids
- Not plug easily
- Be cleaned easily
- Produce clear filtrate
- Be not too expensive
- Be thermally resistant
- Be chemically resistant
- Be strong and not wear easily
- Handle filter aids conveniently
- Allow solids to be discharged easily

The following filter cloths meet some of the preceding requirements and are the most common in industrial filtration:

- Canvas
- Polyamide
- Polypropylene
- Monofilament fiber

Today, most sugar factories use polypropylene and polyamide filter cloths. The filter cloth made from polyamide meets all the general requirements. It is not resistant to hydrochloric acid stronger than 3% (used for cleaning), and is more expensive than filter cloth made from polypropylene. The filter cloth made from polypropylene meets all the general requirements but one: it is not as strong as polyamide.

FILTER AIDS AND SETTLING AIDS

Filter aids are materials that add strength to particles and prevent their breakup. They are usually used when feed contains slimy or very fine solids that can quickly plug the filter pores. Filter aids consist of porous particles. The following types of filter aids are used for filtration:

- Diatomaceous earth
- Perlite
- Diatomite

Diatomaceous earth is a form of silica, processed above 800°C with different particle sizes (5 to 15 microns). Many suppliers offer this product under different trade names.

Settling aids (some call them **coagulants** or **flocculants**) are used in clarifiers to get closer the molecules of precipitates to each other. This increases the density of the solids and their settling rate, improving the clarity of the overflow. Because an increase in the settling rate is desirable, the use of settling aids is common in sugar factories. Settling aids agglomerate the solids (attach to the particles, producing larger and more dense solids that settle faster) in a process called **agglomeration**. The charge carried by the polymer is responsible for the agglomeration of suspended particles. During agglomeration, particles opposite in charge are adsorbed to each other, creating a neutral particle. The neutral particles reject each other, so they remain separate and settle faster. Settling aids are divided into two groups:

- Inorganic coagulants, such as aluminum sulfate, ferric sulfate, and ferrous sulfate
- Water-soluble polymer coagulant, such as acrylamide derivatives, polyethylene oxide, and polyamine polycondensation polymers

Both types of coagulants are known as polyelectrolytes because they have characteristics of both polymers and electrolytes (substances that conduct electricity). Polymer coagulants are water-soluble and have a very high molecular weight (*MW*) compared to inorganic coagulants. The MW of a coagulant may be pictured by its chain length (*the greater the MW, the greater is the chain length and the greater is the agglomeration ability*). A longer chain length enables the attachment of a larger number of particles, improving the settling process.

OPERATING PROBLEMS

Some costly **problems** can be avoided in the sedimentation and filtration operations.

Low Settling Rate in the Clarifier

The optimum settling rate for the best settling of a first-carb juice, produced from normal-quality beets, is 4 to 6 cm/min (7 to 10 m/h or 1.6 to 2.4 in./min). A settling rate below 2 cm/min (1.2 m/h or 0.5 in./min) is low, which slows down the rest of the factory. In addition, it produces an unclear overflow. The following are causes of a low settling rate:

- **Processing low-quality beets**
- **High juice recirculation rate during the first carbonation**
- **Undercarbonated juice:** When juice is gassed insufficiently, it has high alkalinity and pH (above 0.11 alkalinity and 11.1 pH) and a low settling rate.
- **Overlimed juice:** A settling problem may occur if lime is used more than usual (more than 2.5% CaO on beet) when normal beets are processed.

- **Insufficient use of settling-aids:** Use of about 0.0008% OB settling aid is recommended.
- **Low juice temperature:** With normal operations, the juice during first carbonation has a temperature of about 90°C. By the time the juice gets to the clarifier, its temperature is down to about 85°C, which is optimum for a good clarification operation. When a breakdown happens, the juice temperature might become lower than normal, causing settling difficulties. In addition, a low temperature may increase the microbial activities and sucrose inversion in the clarifier.
- **Insufficient clarifier capacity:** A beet-sugar factory needs a clarifier with 60 to 90 m^2 (650 to 970 ft^2) to handle the first-carb juice produced (each day) from 1000 t of beets. Such a clarifier has a retention time of about 60 minutes. A factory with an underdimensioned clarifier may suffer more sugar loss during the processing of damaged beets.

Low Filtration Rate

The usual causes of a low filtration rate in thickening filters follow:

- Low pressure
- Damaged filter cloth
- Incomplete discharge of the residual solids from the filter
- Filtration area covered with the filter cloth in the wrong direction
- Insufficient filter capacity (e.g., for filtration using thickening filters, about 50 m^2 filtration area is required for processing juice from 1000 t of beet per day)

Clogging Filter Cloth of Filter Presses

The usual causes of a clogged filter cloth in the filter presses are as follows:

- Damaged beets being processed: Damaged beets contain more colloids than is the norm, so the filter cloth is apt to clog quickly.
- The lime use is high: When normal beets are processed, the lime should not exceed 2.5% on beet.
- High ammonia content in condensate: If the condensate used for desweetening cake in the filter presses contains a high amount of ammonia, the filter cloth will become stiff and eventually clogged with calcium carbonate.

CHAPTER
3

SECTION 12

STEAM AND POWER PRODUCTION

SUBSECTIONS

- Steam
- Boiler
- Boiler Operations
- Boiler Fuel
- Boiler Feedwater
- Boiler Water Treatment
- Boiler Efficiency
- Steam Turbines
- Operating Problems

ABOUT THIS SECTION **This section** explains **Station 12** (Steam and Power Production) of sugarbeet processing. Station 12 (see Figure 3.12.1) mainly consists of boilers, turbines, the condensate-supply tank, and boiler fittings and accessories. In sugar factories, steam is produced in boilers and used mainly in the evaporation station to concentrate thin juice and in the steam turbines to produce electrical power in powerhouse. When steam is used in the evaporation station, it creates vapor, which is used by the heat exchangers of the factory (e.g., the crystallization and the diffusion station, and juice heaters). Part of vapor condenses into condensate, which returns to the boilers as boiler feedwater and to other places in the factory.

Beet-Sugar Handbook, by Mosen Asadi
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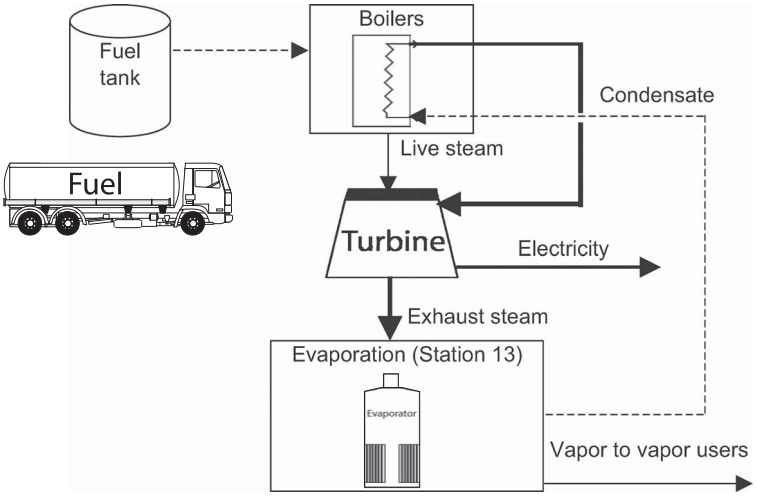


Figure 3.12.1 Flow diagram of steam production (Station 12)

Steam production in the boiler house and its efficient use in the evaporators, turbines, and heat exchangers are of great importance to economy of sugar production. Today, sugar factories try to maintain the steam consumption at about 155 kWh and the power consumption at about 33 kWh per ton of beet. This creates a consumption ratio (heat-to-power ratio) of about 4.7 to 1 between steam and electric power. With the recent increase in fuel costs since 2002, more effort has been applied toward the creation of a more energy-efficient sugar factory.

STEAM

Steam is water in its vapor state produced in a boiler. Heat energy is used to convert water to steam. The state of steam is expressed by either its pressure or its temperature (both functions are dependent on each other). When pressure in a boiler increases, the following increase:

- The boiling point (*BP*) of water
- The temperature of steam
- The enthalpy of steam

Properties of steam, such as the pressure for saturated and superheated steam, can be found from the **steam table** (Table A.10 in the Appendix). For example, after the pressure of steam is determined, the **enthalpy of the steam** (H_V) can be found from the steam table. Then the enthalpy (heat energy) of steam can be used to calculate the quantity of steam required for a particular process.

When a fuel reacts with oxygen from the air, the energy in the fuel is absorbed by the water to produce steam, which is directed to various places in the factory, such as the evaporation station. The fuel can be natural gas, oil, or coal. However, in cane sugar factories, **bagasse** (desugared sugarcane) is used as fuel, so cane factories are almost energy independent. During operation, the boiler's parts are subjected to high temperatures and high pressures, so boilers must be constructed of materials that can resist these conditions.

BOILER

An **industrial boiler** is a closed vessel in which water is evaporated to produce high-pressure steam for the purpose of heating and power. The high pressure increases the temperature of the steam. In other words, high pressure increases the boiling point (*BP*) of water (*at a higher pressure, water boils at a higher temperature*), producing steam with a higher heat energy.

Sugar factories use high-pressure boilers with 30 to 60 bar (440 to 880 psi or 3050 to 6100 kPa) pressure. Today, some sugar factories use 80 bar (1180 psi or 8120 kPa) boilers, which produce superheated steam with temperatures up to 525°C (977°F). A modern turbine working at **live steam** (high-pressure steam from a boiler) of 80 bar and 525°C discharges an **exhaust steam** (steam released from the exhaust of a turbine) with a temperature of about 145°C, which is well above the saturated state and can be used effectively in process (Van der Poel 1998).

The **energy costs** for producing steam are second to raw material in operating costs for a sugar factory. Because the quantity of steam utilized in the process depends on its properties (mainly pressure and temperature), efficient fuel consumption for steam production and for steam and vapor consumption is important to the economy of any sugar factory.

The **boiler house** is a place where one or more boilers, pipes, and auxiliary equipment, such as pumps, tanks, and the steam turbine or turbo generator, are located. A **boiler foreman** operates the boiler house. The **chief engineer** of the factory supervises the boiler house, the evaporation station, the steam distribution, and the auxiliary equipment related to steam production. The person in charge must be familiar with the boiler, the auxiliary equipment, and the steam distribution system in the factory. (In the event of an emergency, *there is no time to review print layouts or trace pipes*.)

Safety is a main concern in operating the boiler house. Therefore, sufficient training of boiler operators and supervisors is of the most importance. The following are a few safety tips:

- The person who operates the boiler house must understand the principles of safety.
- Safety precautions should be applied during the startup and shutdown of boilers.
- Procedures for important situations (e.g., sugar in the boiler) must be documented.
- Procedures for boiler operation (e.g., start and stop of a boiler) must be written.
- Emergency procedures for boiler operation or outages must be documented.

In a sugar factory, steam is mainly used in the following:

- Evaporators to concentrate thin juice and produce vapor
- Steam turbine generator (turbo generator) to produce electrical power
- Steam pulp dryer (high energy costs may make this the standard in pulp drying)
- Sugar-end (note that in modern factories, vapor from evaporators is used in sugar-end)

Live steam (high-pressure steam from the boiler) is directed into the steam header, where it divides into branches that go to the steam turbine and pressure-reducing station. From the pressure-reducing station, the steam divides to feed the evaporation station and is also directed to steam-driven equipment, such as pumps and compressors.

In the evaporation station, the steam is used to concentrate **thin juice** (the product of the purification station). The vapor (low-pressure steam) that is produced is used in other areas

of the factory such as in the crystallization process, the diffusion station, and juice heaters. Vapor from the evaporators condenses into a liquid state, called **condensate**, and returns to the boilers as **boiler feedwater** and to other places in the factory. (Note: Usually only the condensate produced from the second effect of the evaporators is used as boiler feedwater.) Condensate from the third through final evaporator effects (bodies), from sugar-end, and from the process heaters is used for sugar washing, diffuser supply water, cooling water, and other condensate users.

As steam expands and cools in the turbine operation, it gives up some of its **heat energy** to drive the turbine. The rotor of the turbine is connected to the **generator**, which generates power for the factory. Steam released from the exhaust of turbine, known as **exhaust steam**, is sent to the evaporation station.

The combination of steam and power production to use in the process of a factory is known as **cogeneration of heat and power** (CHP). Facilities that produce electricity and steam are called **cogenerators**. In a cogeneration operation (see Figure 3.12.2, case A), live steam is used to drive turbines to produce electricity and exhaust steam.

During the beet campaign, cogenerated electricity is mainly used in the process, but any surplus (excess) electricity is sold to the **public grid** (network). Some factories do not have the turbine to generate their own electrical power and prefer to buy their entire electrical needs from power plant companies. This is because in some countries, power plants offer electricity at a lower price than the sugar factory can produce. Interest in **cogeneration** has increased recently as the cost of fuel and electricity has steadily increased.

During the beet intercampaign (when boilers are shut down), almost all sugar factories buy some electricity, called **standby power**, from local power plants for their needs. During intercampaign, power is needed for equipment maintenance and sugar warehouse activities such as bulk-sugar handling, packaging, and the production of **specialty sugars** (see Section 20 of this chapter).

After steam has released its heat, it **condenses** (turns back to water). The condensate is pure distilled water and is returned to the boiler room to be reused as boiler feedwater. Almost 1 kg of water is required to make 1 kg of steam. For example, a boiler that produces 80 t of steam per hour needs about 80 t of water per hour. In case of condensate loss, makeup water is used

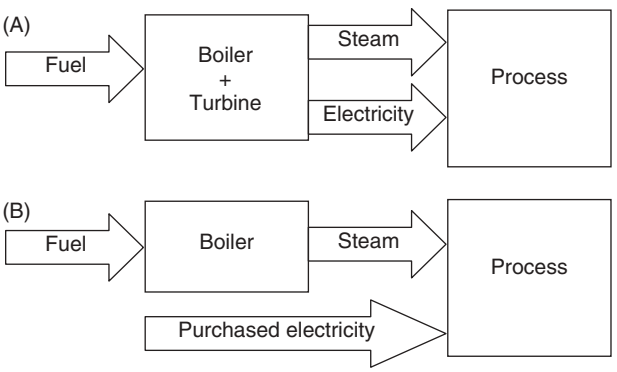


Figure 3.12.2 Factory with cogeneration (case A) and a factory that buys electricity (case B)

to compensate for the loss. (Note: To prevent scale buildup, the **makeup water** should be **softened** before it is used in the boiler.)

Before you start to learn the details of boiler operation, you should know the following necessary terms:

- **Heat** (Q) is a type of **energy** (the ability to do work) created by molecules in motion in a substance. Heat is measured in units such as **joules** (J, rhymes with *tool*) or **calories** (cal).
- **Sensible heat** is the heat that changes the *temperature* of a substance, but not its *state*. For example, as the vapor is used in a heat exchanger, its sensible heat is used to warm up juice from 50 to 90°C (the water in the juice does not change to another state).
- **Latent heat** is the heat that changes the *temperature* and *state* of a substance (the substance can be a gas, a liquid, or a solid). For example, as the steam is used in an evaporator, its latent heat is used to evaporate water from the juice (the water in the juice changes to another state, vapor). For water, the boiling temperature is 100°C (212°F), and the amount of extra heat needed to effect the change (latent heat) is 2256 kJ/kg (970 Btu/lb). 1 Btu/lb = 2.33 kJ/kg. (**Btu** is the abbreviation for a British thermal unit, and kJ is the abbreviation for kilojoule.)
- **Enthalpy** (total heat content) is the sum of sensible and latent heat. In other words, sensible heat is the enthalpy of liquid (or condensate), latent heat is the enthalpy of vapor, and **total heat content** is the enthalpy of steam. **Enthalpy** (H) is important because the difference in enthalpy (ΔH) measures the quantity of heat energy gained or lost by a system.
- **Temperature** (the measurement of hotness or coldness of an object) is expressed in Celsius (°C), Fahrenheit (°F), or Kelvin degrees (K). Refer to the Appendix for temperature conversions.
- The **temperature** of steam depends on its pressure. At atmospheric pressure (1 bar or 14.7 psi), water boils at 100°C (212°F). When pressure increases to 40 bar (588 psi), water boils at 250°C (482°F), and the steam temperature is also at 250°C. An increase in the boiling point of a solution related to its pressure and concentration is called **boiling-point elevation** (*BPE*).
- **Pressure** (the force acting on a unit of area) is measured in kilopascal (**kPa**) or **atmosphere** (same as bar) in the metric system and **pounds per square inch** (psi) in the British system. Because Pa is small, kPa and MPa (megapascal) are often used; 1 MPa = 10^3 kPa.
- **Atmospheric pressure** (the force with which the atmosphere presses down on a given surface area) is equal to 1 atmosphere (atm).
- **Gauge pressure** in the British system is denoted as **psig** (pound per square inch at gauge pressure) and **absolute** pressure is indicated as **psia** (pound per square inch at absolute pressure), or simply psi. Gauge pressure is the measurement of pressure relative to atmospheric pressure. Absolute pressure is the measurement of pressure relative to an absolute vacuum (zero pressure). Gauge pressure can be converted to absolute pressure:

$$\text{psi} = \text{psig} + 14.7$$

- **Power** (the rate of doing work) is measured in **kilowatt** (kW) or **horsepower** (hp).
- **Boilers** are rated in horsepower (hp), which can be roughly estimated by the number of heating surfaces. In boilers, 1 m^2 (10 ft^2) is equal to about 1 hp (0.75 kW).

- **Boiler horsepower** is equal to the evaporation of 15.7 kg (34.5 lb) of water per hour. This equals 15.7 × 2256 = 35420 kJ per hour, or 34.5 × 970 = 33465 Btu per hour. 2256 and 970 are the latent heat of water in kJ (kilo Joules) and Btu (British thermal units). This means that a boiler with 2000 hp can produce 2000 × 15.7 = 31400 kg (or 2000 × 34.5 = 69000 lb) of steam per hour.

Type of Boilers

Two types of boilers are available:

- Water-tube boilers (water is on the inside and heat is on the outside of the tubes)
- Fire-tube boilers (water is on the outside and heat is on the inside of the tubes)

Water-tube boilers equipped with natural or mechanical draft are the most common type of boilers used in the sugar factories. Water-tube boilers can produce steam with higher steam pressure than fire-tube boilers. Water-tube boilers have straight or bent tubes. Boilers with bent tubes have more heating surface and provide better energy efficiency.

NOTE

Packaged boilers (shop-assembled boilers) are assembled at the boiler factory and shipped to the facility for installation. The production cost of packaged boilers is lower than that of field-assembled boilers (assembled in the facility). Today, most new beet-sugar factories use packaged boilers.

Boiler Parts

Boiler parts consist of **fittings** (attached parts) and **accessories** (detached parts). The main fittings of a boiler are the following:

- **Furnace:** For combustion of fuel and air
- **Burner:** For mixing fuel and air to feed them into the furnace in cofiring boilers (oil-fired and gas-fired boilers). The advantage of burning the least expensive fuel is important.
- **Bunker** (stocker): For feeding coal into the furnace in coal-fired boilers
- **Safety valve:** For protecting the boiler from maximum allowable working pressure (MAWP)
- **Blowdown valve:** For blowdown
- **Main steam stop valve:** For taking boiler off line
- **Steam pressure gauge:** For showing steam pressure in the boiler (in gauge-pressure unit)
- **Vacuum gauge:** For showing vacuum pressure
- **Water column:** For reading of the water level

The main boiler's accessories are as follows:

- **Feedwater valve:** For controlling the flow of feedwater to the boiler
- **Condensate tank:** For holding condensate (is used as boiler feedwater)
- **Pulverizer:** For grinding and pulverizing coal in boilers using pulverized coal

- **Feedwater pump:** For pumping feedwater to the boiler (reciprocating, electric-driven centrifugal, or steam-turbine-driven centrifugal pumps are used for feedwater pumping)
- **Feedwater regulator:** For maintaining the amount of feedwater to the boiler (common types are thermo expansion, thermo hydraulic, three-element, and float feedwater regulators)
- **Feedwater heater:** For heating feedwater (feedwater is mostly heated by using heat from the boiler **flue gas**, which is gas burned before entering the stack of the boiler)
- **Feedwater economizer:** For recovery of heat from the flue gas to be used in the feedwater heater for heating the feedwater
- **Air heater:** For heating combustion air to the ignition temperature using the heat of the flue gas (air heater is located between the boiler and the stack)
- **Economizer:** For recovery of the flue gas and adjusting its temperature
- **Fuel oil strainer:** For removing foreign materials from oil in oil-fired boilers
- **Fuel oil heater:** For heating oil (particularly No. 5 and 6 oil) in oil-fired boilers.

BOILER OPERATIONS

Boilers are costly, so close attention must be paid to their operations. In boiler operations, all parts of the boilers and their auxiliaries must be checked at all times, including the following:

- **Water level**
- **Steam pressure**
- **Feedwater temperature:** Keep at high temperature to eliminate oxygen, but too high a temperature can hurt the feedwater pump
- **Burner:** Burner tips, ignition electrodes, and strainers must be checked for cleanness

The startup procedure for boilers is important. The common procedure for starting boilers includes the following:

- **Feedwater check:** The quality of the feedwater must be checked.
- **Fuel preparation:** When coal is used as a fuel, the supply of coal on hand must be sufficient before startup. When fuel oil is used, its supply must be sufficient and must be at the proper temperature before it can be burned. When gas is used, the correct pressure at the burner must be maintained.
- **Slow startup:** Boilers must be warmed up slowly to prevent uneven expansion of refractory and metal parts.

The shutdown procedure at the end of the campaign is also important. Proper shutdown prevents the boiler equipment from being damaged during intercampaign.

NOTES

- **Recognition of problems that may occur in the boiler operation is important. Problems such as the presence of sugar in feedwater must be recognized in the early stages and remedial actions must be taken immediately.**
- **The startup of the boilers at the beginning of the campaign and their shutdown at the end is critical to the operation of the boilers and their life expectancy.**

BOILER FUEL

Fuels are carbon-based chemicals consisting mainly of carbon, hydrogen, oxygen, and sulfur. The presence of sulfur is undesirable. Fuel with a low oxygen content is desired (*the less oxygen a fuel contains, the more energy per mass unit it releases*).

Common fuels used in the boiler are the following:

- Coal
- Oil
- Natural gas

Coal, oil, and natural gas are the remnants of fossils. In the prehistoric past, vast quantities of plants were buried under layers of sediment. High pressure and a lack of oxygen stopped the decomposition of organic compounds and resulted in the formation of coal and petroleum. Natural gas is usually found near coalmines and petroleum wells.

The type of fuel used in the sugar factory boilers depends on the following:

- Fuel price
- Fuel resource availability
- Environmental policies with respect to emissions

Coal has an approximate chemical formula of $C_{135}H_{96}O_9NS$ and a typical heat value of 30000 kJ/kg (13630 Btu/lb). **Heat value** (*HV*, it is also called the **heat of combustion**) is the quantity of heat released by combustion of a mass unit of a fuel.

Coal is classified according to its **grade** (size, heat value, and ash content). These are four different types of coal:

- Anthracite
- Bituminous
- Subbituminous
- Lignite

Lignite (brown coal) is the lowest grade because of its lower *HV*. The *HV* of bituminous and anthracite coal is higher than that of lignite because they have been exposed to a higher pressure, which caused them to lose more oxygen and moisture and become harder. Lignite is used in some sugar factories. Anthracite has a higher carbon and lower sulfur content than other coals. This makes anthracite the most preferred grade of coal, with relatively small leftover deposits.

The bulk density of medium-size coal is about 750 kg/m^3 (47 lb/ft^3). Coal is a dirty fuel physically as well as environmentally. The largest source of SO_2 in the air is produced from the combustion of coal. The unburned particulate matter from coal heaters in past centuries blackened the buildings of many beautiful cities. In addition, if sulfur is not filtered, it can contribute to **acid rain** (atmospheric precipitation as rain, fog, and snow, with a pH less than 5.5). SO_2 can react with water in the air to form sulfurous acid:

$$SO_2 + H_2O \rightarrow H_2SO_3$$

Nitrogen dioxide (NO_2) and nitrogen oxide (NO) produced by coal-burning factories and in automobile engines also contribute to acid rain.

$$3NO_2 + H_2O \rightarrow 2HNO_3 + NO$$

$$2NO + H_2O \rightarrow 2HNO_3$$

(Note that in environmental literature, **NO_2** and **NO** are summed and indicated as **NO_X** [rhymes with box].)

Pulverized coal is also used in some sugar factories. Coal is pulverized by the coal **mills** (pulverizer). Boilers operating with pulverized coal have a higher efficiency than boilers using solid coal. But the installation and operating costs are higher due to the pulverizing operation. Pulverized coal is highly explosive, so special considerations must be made in its handling. In addition, it needs a large furnace volume to complete combustion.

Coal is usually analyzed for percentages of carbon, oxygen, hydrogen, nitrogen, ash, and sulfur. Coal can have 4 to 20% **ash** (incombustible solids). During combustion, the coal ash (**fly ash** or **particulate matter**) is carried by the flue (combustion) gas and is removed by **scrubbing equipment** to prevent its discharge into the atmosphere as an air pollutant. Coal with low sulfur is in demand because of its low sulfuric acid formation during combustion. Sulfuric acid attacks boiler metals. The sulfur in coal also results in sulfur dioxide emission.

Coal is shipped during the intercampaign and stored in sufficient quantities to cover the fuel needs of the factory. Coal must be handled properly to prevent the possibility of combustion. Coal must be used on first-come, first-use basis. The coal temperature is checked at various points in the pile. If the coal temperature increases rapidly, carbon dioxide may be applied to the coal pile by using perforated pipes to decrease the temperature of the coal pile.

Coal, like all fuels, has some ash known as **fly ash** or **particulate matter**, which is defined as incombustible solid matter of the fuel. Coal's ash ranges from 4 to 20%.

Fuel oil is also classified by grades (No. 1 up to No. 6). A typical *HV* of oil is 48000 kJ/kg (21820 Btu/lb). Fuel oil contains mainly carbon and hydrogen with some sulfur, nitrogen, and moisture. Low sulfur content is desired to reduce SO_2 emitted into the atmosphere. The density of oil is important in determination of HV. Heavy fuel oils (No. 5 and 6) have a higher *HV* than light fuel oils, but they should be heated to lower the viscosity to make pumping easier. Fuel oils must not be overheated because they can form sediment, which settles in the tank.

Natural gas is cleanest and least polluting fuel to use in a boiler. But when it is incompletely combusted (when all the fuel is not burned), smoke forms. In addition, natural gas is far more subject to explosion than oil or coal. Natural gas contains methane (CH_4), ethane (C_2H_6), propane (C_3H_8), and small amounts of hydrogen, nitrogen, and carbon monoxide.

The *HV* of fuel is determined by calculations based on the chemical analyses of the fuel:

$$HV = 33878\mathrm{C} + 144460\left(\mathrm{H} - \frac{\mathrm{O}}{8}\right) + 9436\mathrm{S}\ \mathrm{kJ/kg} \tag{3.12.1}$$

$$HV = 14540\mathrm{C} + 62000\left(\mathrm{H} - \frac{\mathrm{O}}{8}\right) + 4050\mathrm{S}\ \mathrm{Btu/lb} \tag{3.12.2}$$

C	% carbon
H	% hydrogen
O	% oxygen
S	% sulfur

(Note: The value oxygen/8 exists because some hydrogen combines with oxygen to form water.) The preceding equation is based on the amount of combustion heat released from each component of fuel:

C = 33878 kJ/kg (or 14540 Btu/lb)
H = 144460 kJ/kg (or 62000 Btu/lb) – 1/8O
S = 9436 kJ/kg (or 4050 Btu/lb)

EXAMPLE 1

Given:
A fuel has the following analyses:

C	80%
O	6%
H	5%
N	4%
S	1%
Ash	4%

Calculate:
The *HV* of the fuel

$$HV = 33878 \times 0.8 + 144460\left(0.05 - \frac{0.06}{8}\right) + 9436 \times 0.01 = 33336 \quad \text{kJ/kg}$$

$$\frac{33336}{2.33} = 14307 \quad \text{Btu/lb}$$

Fuel Combustion in a Boiler

Combustion is the chemical reaction of combustible components of fuel with oxygen to create heat and a flame, as in the burning of a match. Combustion reactions are **exothermic** (energy releasing). Before fuel can burn, it must be heated to the ignition temperature.

As fuel burns, its carbon–hydrogen bonds break and the carbon and hydrogen atoms combine with oxygen molecules (O_2) in the air to produce carbon dioxide (CO_2), water, and heat:

$$C + O_2 \rightarrow CO_2 + \text{heat energy}$$

$$2H_2 + O_2 \rightarrow 2H_2O + \text{heat energy}$$

Sulfur in fuel combines with oxygen to form sulfur dioxide:

$$S + O_2 \rightarrow SO_2 + \text{Heat energy}$$

The oxygen needed for the combustion reaction is provided by air, which must be mixed with fuel in the most efficient way. The air we breathe is a mixture of nitrogen (N), oxygen (O), water (the amount depends on the weather's moisture), and a small amount of **carbon dioxide**

(CO_2). **Dry air** typically contains about 21% (by volume) of oxygen, 78% nitrogen, and 1% carbon dioxide gas. These data are usually used in technical calculations.

The reaction of **hydrocarbon compounds** (compounds containing carbon and hydrogen, e.g., methane and ethane) with oxygen is highly **exothermic** (energy releasing). The *HV* of ethane (C_2H_6) is high (about 52000 kJ/kg), meaning that when 1 kg of ethane burns, 52000 kJ of heat are released. The combustion of ethane (the main component of natural gas) can be described by the following equation:

$$C_2H_6 + 3/2O_2 \rightarrow 2CO_2 + 3H_2O + 1560\,\text{kJ Heat}$$

The **combustion air requirement** (CAR) is kg (or lb) of air needed for the combustion of each kg (or lb) of fuel. You can calculate CAR based on the analysis of the fuel. To achieve an efficient combustion, some **excess air requirement** (EAR) above the theoretical amount is needed. Without sufficient oxygen, the combustion is incomplete, causing the formation of carbon monoxide (CO), which is an odorless, tasteless, and highly **toxic gas**. Usually, an excess (surplus) air coefficient of 1.3 is used. Theoretically, to burn 1 kg of fuel oil, 13.4 kg of dry air are needed. Therefore, the practical need is equal to $13.4 \times 1.3 = 17.4$ kg of dry air per kg fuel. For the combustion of each cubic meter of natural gas, 10.2 m^3 air is needed. A coefficient of 1.3 brings this amount to 13.3 m^3 of air.

The quality of the combustion process can be evaluated based on the composition of the flue gas. The quality of combustion can be judged by the composition of the flue gas:

- High carbon monoxide is an indication of incomplete combustion
- High oxygen (more than 4%) is an indication of excess air

The following are used for analyzing flue gas:

- **Fyrite analyzer:** Measures the percentage of oxygen and carbon dioxide
- **Orsat analyzer:** Measures the percentage of oxygen, carbon dioxide, and carbon monoxide

NOTES

- **Modern sugar factories are equipped with an economizer. The economizer recovers heat from boiler flue gas (combustion gas) and exhaust gas (hot gas from the steam generator), and transfers it to heat users. Heat users use the recovered heat to heat, for example, boiler feedwater. This saves energy consumption of the factory considerably. Boilers produce 2.2 kg (1.6 m^3) of the flue gas from evaporating 1 kg of water to steam. This amount of the flue gas contains about 300 kJ of heat.**
- **A boiler furnace is similar to the furnace in a pulp dryer.**

Boiler Draft

The **boiler draft** is the movement of air from higher pressure to lower pressure to be used in the combustion of fuel. A difference in pressure is required to get air into the combustion chamber of the furnace. The pressure can be applied naturally or by force.

In **natural-draft boilers**, the rising property of warm air is used to move the flue (combustion) gas out of the stack. As air warms, it becomes lighter and moves upward; colder air is heavier and moves below the warmer air (*a container filled with heated gas weighs less than a*

container of cool gas). The movement of warmer and colder air creates a draft. (Home fireplaces use natural draft to provide air for combustion.) In natural-draft boilers, the efficiency of draft is limited by the stack height (*the higher the stack's height, the stronger is the draft*) and the temperature difference between the inside and outside of the stack. In these boilers, the outlet and the inlet air dampers control draft to supply air to the furnace and discharge the flue gas from the stack.

In **forced-draft boilers**, fans are used to move the flue gas. New sugar factories no longer use tall stacks. Instead, the boilers are equipped with draft fans. In forced-draft boilers, the draft is controlled by the speed of the **fans** and the adjustment of the air dampers. Fans are located either in front of the boiler to force air into the furnace or between the boiler and stack to discharge the flue gas from the stack. **Combination-draft boilers** use both natural draft and fans.

BOILER FEEDWATER

Boilers need a considerable amount of water for their operations (25 to 35% OB). As mentioned, a boiler with 2000 hp can produce:

$$2000 \times 15.7 = 31400 \text{ kg of steam/hour}$$

or

$$2000 \times 34.5 = 69000 \text{ lb of steam/hour}$$

Such a boiler needs a minimum of 31 400 kg of water per hour. The main part of this water is supplied by **condensate** (steam that has lost its heat and has been converted to water), which returns to the boilers as feedwater. Pure condensate has the following properties:

- Low impurities
- Low conductivity
- Suitable pH (between 7 to 10)
- Low hardness (close to 0 ppm)
- Free of oxygen (oxygen creates corrosion in the boiler tubes)

In sugar factories, condensate is produced from different sources, and all are not appropriate for use as feedwater in boilers. This is due to the presence of impurities in the condensate (particularly sugar) deposited on the boiler tubes. Sugar at high temperatures decomposes and starts to deposit on the boiler tubes, and this can damage them considerably (see the Operating Problems section later in this section).

In most sugar factories, feedwater is supplied mainly from the condensate produced in the second-effect evaporators. When there is not enough condensate, **makeup water** (usually softened city or well water) is added to the boiler to replace the water that has been lost by blowing the boiler or through leaks in the system. The raw water has to be softened before being used as makeup water because the hardness of water is often high and can lead to excessive boiler blowdown and waste of energy. **Blowdown** is the process of opening the boiler's blowdown valve for a short period to remove the suspended materials from the boiler. The best time to blowdown is when the boiler is under a light load. Later in this section, there will be more talk about water softening.

Feedwater from the feedwater tank is heated in heaters before being pumped to the boiler. The temperature of the feedwater entering the boiler is important to the boiler's energy consumption. If the feed temperature is at high, all the heat is available for steam production. If the feed temperature is low, part of the heat has to be used for heating the water to its boiling point. In addition, cold water is a **thermal shock** to boiler metal and increases wear and tear on the boiler.

Evaporators and most heat exchangers are equipped with a **steam trap** that allows condensate, but not steam, to be discharged. Most trap systems operate on a gravity principle. The condensate receiver draws the condensate by gravity out of the steam chest to the primary condensate tank. From the primary tank, the condensate is pumped to the main feedwater tank and then to the boiler.

NOTES

- **Sugar detection equipment is usually installed to continuously check the presence of sugar in each stream directed to the boiler feedwater tank.**
- **Condensates from juice heaters usually contain traces of sugar. This is because in the heaters, the juice is often at a higher pressure than the vapor, causing some juice to get into the steam chamber and contaminate the condensate.**

Feedwater Softening

Boiler feedwater mainly consists of condensate from the boilers and makeup water. Raw water (well or city water) is hard (contains minerals) and cannot be used as makeup water because most minerals are scale forming at high temperatures. If hard water is mixed with condensate, minerals start to build up in the boiler, overheating and eventually burning out the boiler tubes.

The salts that form the most scale are calcium (Ca) and magnesium (Mg) salts, known as **hardness** (limesalts). Hardness deposits on the boiler tubes, reducing heat transfer. In addition, raw water contains oxygen and noncondensable gases, such as ammonia, that create **corrosion** (a chemical process caused by oxygen) in the heating tubes of the boiler. Therefore, before hard water enters the boiler, it is softened in one of the following ways:

- By adding chemicals
- By ion-exchange resin

In **water-chemical softening** (caustic-soda softening), caustic soda (NaOH), soda ash (Na_2CO_3), or a combination of both is used to precipitate Ca and Mg salts. In the **combination chemical method** (lime-soda process), liquid caustic soda and powdered soda ash are mixed with water to precipitate hardness. The precipitate is removed from the water by filtration. Cold chemical softening is performed at room temperature (about 20°C), and the hot process is performed at about 90°C, using steam to heat the water.

Water softening by ion-exchange resin belongs to a separation group named **ion chromatography**. In this process, calcium (Ca) and magnesium (Mg) ions in water are exchanged for nonscaling sodium (Na) and held on the surface and pores of an ion-exchange resin. When resin gets exhausted with Ca and Mg ions, it is regenerated with a solution containing Na, such

as ordinary salt (NaCl, sodium chloride) solution. Water softening is similar to thin-juice softening (see Chapter 6).

In addition to hardness, boilerfeed water should be checked for the presence of oxygen and for pH. Oxygen and noncondensable gases are removed from the boiler feedwater by heating the water in an open heater or in a deaerating heater. The pH of boiler feedwater should be maintained between 8 and 10. Caustic (sodium hydroxide) is usually added to water with a low pH to raise the pH value.

BOILER WATER TREATMENT

Boiler water (water that circulates in the boiler drum and tubes) is treated by adding chemicals directly into the boiler to condition its properties. Water samples from the boilers are taken regularly to determine their condition and the necessary treatments.

To prevent scale formation on the tubes, sodium hydroxide (NaOH) is added to the boiler water to change the leftover hardness (scale-forming salts) into a precipitate, which accumulate at the bottom of the boiler. The precipitate is then removed by blowing down the boiler.

Boiler water hardness is analyzed using EDTA.$2H_2O$ (simply EDTA). EDTA (ethylendiamine tetraacetic acid dihydrate) is a **chelate** (a neutral molecule that can form a bond to a metal ion) and bonds to calcium and magnesium salts to form a complex ion. (See the following Notes for more use of EDTA.)

The oxygen in boiler water **corrodes** (rusts) the heating tubes. Adding an **oxygen scavenger** eliminates the leftover oxygen in boiler water. Oxygen scavengers are chemicals, such as **sodium hydroxide** (NaOH), **sodium sulfate** (Na_2SO_4), and **sodium phosphate** (Na_3PO_4), that combine with oxygen to convert it to a harmless suspension that is removed through the blowdown line.

The alkali and ferrous salts and silica (SiO_2) content in boiler water is important. The presence of these substances in steam is dangerous for steam turbine operation because they can precipitate on the turbine blades. The allowable content of SiO_2 in boiler water depends on the boiler pressure because its solubility increases sharply at higher pressures. At 80 bar pressure, the maximum allowable SiO_2 content is 5 ppm.

An alkalinity test is used to determine the alkalinity of boiler water. The correct alkalinity prevents scale formation. The P-alkalinity test (**P** is for phenolphthalein) of boiler water should be 250 to 350 ppm. A M-alkalinity (**M** is for methyl purple) test indicates the total alkalinity.

NOTES

- **By chemical treatment of the boiler water and proper blowdown, boilers can operate without difficulties. Continuous blowdown is in common use in most sugar factories because it gives better control than intermittent (noncontinuous) blowdown. In continuous blowdown, heat is recovered by flashing the blowdown line.**
- **Outside companies specialized in the operation and chemical treatment of boilers are hired by many sugar factories to oversee boiler operation, startup, and shutdown. They also help the in-house boiler operators perform corrective actions during an emergency. After each campaign, they usually monitor boiler tubes for scale deposition with special cameras.**
- **EDTA can be used as a scavenger to remove the calcium salts from the thin juice and clean the evaporators. Such a treatment is costly and is not practiced in sugar factories.**
- **EDTA is also used as a reagent in analyses of hardness in water (see Chapter 11).**

BOILER EFFICIENCY

Boiler efficiency (*BE*) is the ratio of heat output to heat input (heat value of fuel burned). In other words, *BE* is the percentage conversion of fuel to steam. In sugar factories, boilers absorb approximately 80 to 90% of the heat value available in fuel. Because the feedwater temperature and the heat value of fuel in a given factory are constant, the following simple equation can be used for boiler efficiency:

$$BE = \frac{W_S}{W_F} \tag{3.12.3}$$

W_S Amount of steam
W_F Amount of fuel

(Note: This equation indicates the **evaporation rate** rather than boiler efficiency.) Preparing data to use the equation is easy because sugar factories have a steam flow meter for recording steam production and another flow meter for fuel consumption (for oil-fired or gas-fired boilers) or a scale (for coal-fired boilers).

EXAMPLE 2

Given:
A factory with 3 gas-burning boilers and without turbine

Beets processed	8000 short t/day
Steam produced	2420 short t/day
Natural gas usage by boilers	54000 therms/day

Calculate:
Boiler efficiency (*BE*)
% Steam on beet (% OB)

$$BE = \frac{W_S}{W_F} = \frac{2420 \times 2000}{54000} = 89.6 \quad \text{lb Steam/therm of gas}$$

$$\text{Steam} = \frac{2420}{8000} \times 100 = 30.3\% \quad \text{OB}$$

EXAMPLE 3

Given:
A factory with 2 oil-burning boilers and no turbine

Beets processed	8000 t/day
Steam produced	1770 t/day
Oil usage by boilers	9300 L/day
Oil density	1.0 m^3/t

Calculate:
Boiler efficiency (*BE*)
% Steam on beet (% OB)

$$BE = \frac{W_S}{W_F} = \frac{1770}{9.3} = 190.3\,\text{t} \quad \text{Steam/m}^3 \text{ of oil}$$

$$\text{Steam} = \frac{1770}{8000} \times 100 = 22.1\% \quad \text{OB}$$

Energy losses in boilers occur because of a number of reasons:

- Stack
- Moisture in air
- Incomplete combustion
- Low-temperature feedwater
- Low-temperature combustion air
- Loss during steam transportation (poor insulation of the pipes)

STEAM TURBINES

Steam turbines convert the **heat energy** (enthalpy, H) of steam to **kinetic energy** (K_E). A system containing energy can perform work. In turbine operation, the amount of work performed depends on the heat energy and the velocity (speed) of steam used in the turbine.

When steam passes through a **narrow opening** (throttle point), pressure is converted into kinetic energy (K_E energy due to the motion of the object) with high velocity. The K_E acts as a force to turn the turbine rotor at a high rotational velocity (up to 10000 RPM), which drives the generator. A hydraulic governor maintains a constant speed of the rotor. A magnetic field and a rotating wire loop in the generator produces electricity. The output power (P_O) of the turbine at steady-flow condition is given as:

$$P_O = m_S(H_1 - H_2) \tag{3.12.4}$$

where m_S is the mass flow of the steam passing the turbine and H_1 and H_2 are enthalpy (the heat content of the steam) of the steam at inlet to the turbine and outlet of the turbine.

As steam passes through a steam turbine, the steam volume increases, and its pressure decreases when it leaves the turbine. Steam leaving the turbine, called **exhaust steam**, is mainly used in the evaporators. Steam turbines are used to drive:

- **Electric generators** for producing electricity
- **Auxiliary equipment** such as pumps and compressors

In an impulse turbine, energy acts in a forward direction on a blade mounted on a wheel. Impulse steam turbines may contain few sets of wheels. To increase the velocity in each stage, steam is directed through a nozzle, causing the steam to expand and increase in velocity. An impulse turbine consists of the following main parts:

- Shaft gland to prevent steam leakage
- Nozzle to increase the pressure of steam
- Shaft to support mounted wheels with blades
- Condenser to reduce the back pressure of the turbine by reducing the water rate
- Governor to control the turbine speed when the speed of the steam changes
- Rotor to make centrifugal force, rotating on a layer of lubricating oil
- Lubrication system to prevent rotor from friction and wear

OPERATING PROBLEMS

A number of **problems** can occur in boilers operations.

Sugar in Boiler Feedwater

Even a small amount of sugar in condensate (boiler feedwater) can damage boilers. At high temperatures, sugar breaks down into acids, causing foam, corrosion, and **deposit** (scale) inside the boiler tubes. This damages the boiler and its accessories considerably. In serious cases, the boiler must be shut down for corrective actions.

Inline instruments such as **conductivity meter** or **flame photometer** are installed in the feedwater line and can detect even a trace of sugar in the boiler's feedwater. Boiler feedwater is tested for sugar in the laboratory hourly, and even more frequently when a trace of sugar is detected. (See Chapter 11 for information on the **Alfa-naphtol test** for sugar detection in boiler feedwater.)

Even with the best preventive measures, sugar does sometimes get into feedwater. When sugar is in the condensate, small doses of caustic soda can be added to the feedwater to neutralize its acidity. (Note: A high rate of caustic addition creates foaming in the boiler, causing carryover of impurities into the steam and contamination of the steam system.) Therefore, you must apply defoaming agent.

Sugar usually gets to condensate because of the following reasons:

- **Leak in heating tubes:** When juice has a higher pressure than vapor in the heat exchangers, juice can get to the heat section and contaminate it with sugar.
- **High juice level in the evaporators:** If you keep the juice level in the evaporator at a high level, juice bubbles rise to the top, entering the steam chest of the next effect evaporator, causing the presence of sugar in condensate.
- **Aggressive boiling in evaporators:** Aggressive boiling causes the same situation as when the juice level in the evaporators is kept at a high level.
- **Foaming in the evaporators:** Excessive foaming can carry along sugar, causing contamination of the condensate.

High Hardness in Feedwater

Hardness (the scale-forming dissolved salts of calcium and magnesium) starts to settle at high temperatures. The hardness in feedwater should be kept as close to 0 ppm as possible. High hardness in water discharged from the makeup water softener is the most likely reason for high hardness in feedwater. Monitoring makeup water hardness allows better control of water-softening operations, particularly its regeneration intervals.

High Oxygen Content in Boiler Feedwater

Water absorbs nitrogen, oxygen, and carbon dioxide from air. Nitrogen does not damage the boiler's tubes, but oxygen and carbon dioxide are corrosive and can damage the boiler's drum and tubes.

The solubility of oxygen in water decreases with increasing temperature, so water loses oxygen when its temperature increases. The presence of oxygen in the boiler feedwater happens when the temperature of the feedwater heater is not enough to raise the temperature of the feedwater sufficiently. Heating the water in the feedwater heater and then deaerating it in a deaerator chamber by good venting to the atmosphere removes oxygen and noncondensable gases and solves this situation. Heating feedwater also helps steam economy.

Oil in Feedwater

Oil can get to the boiler and cause a problem for boiler operations. Oil mostly gets to the feedwater from steam equipment, such as pumps, or from equipment in which steam and water are in close contact, such as an open feedwater heater. Some steam equipment have an oil baffle or filter bed to trap oil.

Feedwater Pumping Difficulties

Sometimes the feedwater pump cannot deliver condensate to the boiler. This condition is called **steam bound**. Following are two causes of a pump becoming steam bound:

- A rise in water temperature from steam pressure that is too high.
- Rapidly changing feedwater temperature from an inconsistent steam load.

CHAPTER
3

SECTION 13
JUICE EVAPORATION

SUBSECTIONS

- Evaporation Theory
- Evaporation in a Sugar Factory
- Multiple-Effect Evaporation
- Heat Exchangers
- Types of Evaporators
- Concentrators
- Evaporation Control and Energy Saving
- Steam-Pressure Reduction and Vapor Compression
- Scale Formation in Evaporators
- Evaporator Cleaning
- Material and Heat Balance
- Operating Problems

ABOUT THIS SECTION **This section** familiarizes you with **Station 13** (Juice Evaporation) of sugarbeet processing. Station 13 (see Figure 3.13.1) is the last station of the beet-end and includes the evaporators and juice heaters. The evaporation station can be viewed as the **heat center** of the factory because it serves the sugar factory in three ways:

- Concentrates thin juice
- Produces condensate for boilers
- Produces vapor and condensate for heat users

Energy cost is the second major operating cost in sugar production (raw material is the first). Energy prices have been increasing considerably, so steam production (in the boiler house), its consumption (in the evaporators and turbines), and vapor distribution to heat users become more important in sugar production. Beet-sugar factories consume 20 to 30% steam on beet (OB) for evaporation and heating purposes. (In some modern European factories, steam consumption has been as efficient

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as 10% OB.) This is an efficient operation considering the fact that the amount of water to be removed by evaporation in a beet factory is about 95% OB (75% of beet is water and about 20% water is added during the diffusion process) and that a single-effect evaporation uses about 1 kg of steam to evaporate 1 kg of water. This is why sugar factories use **multiple-effect evaporation,** in which 1 kg steam entering the first effect can evaporate as many kilograms of water as there are effects in the evaporation station.

The main goal of this section is to familiarize you with the principle of evaporation and the optimization of its operation. Most of the discussion is directed toward multiple-effect evaporation and its capacity and economy. Related subjects such as material and heat balances, and operating problems are also discussed.

History: In 1850, F. Robert developed the first **vertical-tube evaporator**. Soon later, N.Rillieux (a French sugar technologist) developed the **multiple-effect evaporation**.

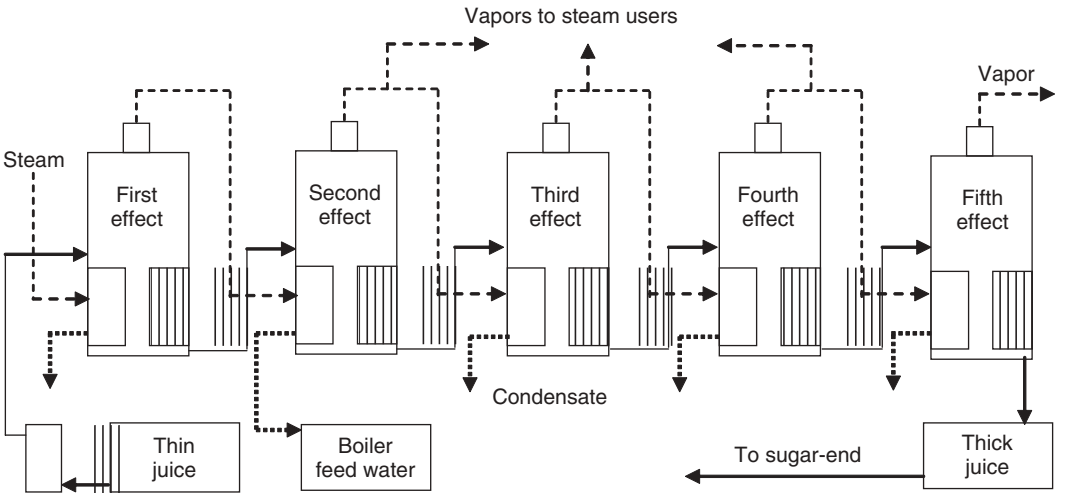


Figure 3.13.1 Flow diagram of a five-effect evaporation (Station 13)

Evaporation (vaporization) is the process of concentrating a solution by boiling to convert some of the liquid to vapor. The **temperature difference** between the heating medium (steam) and feed is the driving force (cause) of evaporation. This definition includes two classes of evaporation:

- Heating evaporation (conversion of liquid to vapor by heating under normal pressure)
- Flashing evaporation (conversion of liquid to vapor by heating under vacuum)

In the sugar factory, flashing evaporation is used to obtain a lower boiling temperature of the juice, which decreases the required heat. Low pressure decreases the **boiling point** (*BP*) of water (*at a lower pressure, water boils at a lower temperature*), resulting in a heat-efficient operation (saves energy). To save more energy, heat exchangers preheat the feed before it is introduced to the evaporators. For evaporation, **heat** (flow of energy from a hotter object to a colder object) is needed to convert molecules of the solution to the vapor state. The equipment that uses heat to concentrate a solution is called an **evaporator**.

Boiling is a rapid form of evaporation that occurs when a liquid reaches its *BP*. The difference between evaporation and boiling is that evaporation can occur without reaching the *BP* (water in the container evaporates without reaching its *BP*), but *boiling occurs only when the liquid reaches its BP*.

Condensation is the opposite of evaporation. During evaporation, vapor forms and then condenses back to a liquid, called **condensate**.

$$\text{Liquid} \xrightarrow{\text{EVAPORATION}} \text{Vapor} \xrightarrow{\text{CONDENSATION}} \text{Liquid}$$

In the sugar factory, water is evaporated from juice in two stations:

- Evaporation station
- Crystallization station

In the evaporation station, thin juice with about 15% dry substance (*DS*) and about 8.8 pH is concentrated to produce a thickened juice with about 60% *DS* and about 8.7 pH, which is called thick juice. Thin juice consists of nonvolatile solutes (sucrose and nonsucrose substances) and a volatile solvent (water) (see the Notes below).

NOTES

- **A solute is a substance (in our case, sugar and nonsugars) dissolved in a solution, and a solvent is the liquid (in our case, water) in which solute (solutes) is dissolved. During evaporation, only the solvent (water) evaporates; solute (sugar and nonsugars) does not. This is the reason why the purity of the juice does not change during evaporation (purity is not relevant to water content), assuming no decomposition of sucrose occurs.**
- **Evaporation stations used to be installed inside the main building of the factory. Today, in some newly built or reconstructed factories one or more evaporators or the entire evaporation station is installed in the open area. This is possible because of a high insulating technique used by factories in recent years.**

Before you continue to study the details of the evaporation station in a sugar factory, it helps to know some definitions of evaporation in general.

Atmospheric (barometric) pressure works with gravity on an object and depends on altitude. (Atmospheric pressure is greatest at sea level.) For every 5 km (3.1 mile) increase in altitude, the pressure drops by half. At about 100 km altitude, the pressure drops to 0 (vacuum). The boiling point changes with elevation because different elevations apply a different amount of pressure on the solution (*the higher the elevation, the lower is the pressure*). Therefore, the boiling point depends on the applied pressure (*the lower the pressure, the lower is the BP*). The normal boiling point is defined at standard **atmospheric pressure** (760 mmHg = 760 torr = 1 atm = 101.3 kPa = 14.7 psi). At sea level, where the atmospheric pressure is 1 atm (14.7 psi), water boils at 100°C (212°F). Pressure is expressed in the following units:

$$1\,\text{psi} = 6.9\,\text{kPa} = 0.069\,\text{bar}$$

$$1\,\text{atm} = 1.013\,\text{bar} = 101.3\,\text{kPa} = 25.4\,\text{in. Hg} = 760\,\text{mmHg} = 760\,\text{torr} = 14.7\,\text{psi}$$

Gauge (relative) pressure in the British system is denoted as **psig** (pound per square inch at gauge pressure). **Absolute pressure** is indicated as **psia** (pound per square inch at absolute pressure) or simply psi (psi = psig + 14.7).

Boiling point elevation (*BPE*) is the increase in the boiling point (*BP*) of a solution over that of pure water. Therefore, the *BPE* of an impure sucrose solution such as thin juice can be defined as the difference between the boiling point of the juice and the boiling point of water at the same pressure.

During the evaporation of thin juice, the *BPE* of the juice increases as its *DS* content increases, so it must be heated to a higher temperature to come to a boil. For example, a 50% sugar solution boils at 101.8°C (its *BPE* is 1.8°C), but a 60% sugar solution boils at 103.1 (its *BPE* is 3.1°C). (*BP* is *pressure dependent*, but *BPE* is *concentration dependent*.)

Heat (*Q*) is the flow of **energy** (the capacity to do work or supply heat) from a higher-temperature object to a lower-temperature object. Heat is the product of the breaking of chemical bonds in the fuel (oil, coke, coal, or natural gas) in a combustion reaction. *Combustion* is a chemical reaction between components of a fuel with oxygen, which creates heat. Sugar factories use natural gas, oil, and coal in the boilers to produce steam.

Steam tables present the properties of steam, such as vapor pressure, heat of evaporation, and specific heat capacity. You can use steam tables to determine the **enthalpy of steam** (the heat content of the steam) if you know the temperature or pressure of the steam. This information can be then used to calculate quantity of steam and the energy required for the process. The **saturated steam table** is the most common table used for steam calculations. Steam table (Properties of Saturated Steam and Water, Table A.10) is provided in the Appendix at the end of the book.

EVAPORATION THEORY

At room temperature, molecules in a solution are attracted to adjacent molecules by **intermolecular forces** (I_F, the forces that hold molecules together because of chemical bonding). In addition, the molecules are under the effect of atmospheric pressure, which tries to keep the molecules together.

For a molecule to escape (evaporate) from a solution, two conditions are required:

- The molecule must be at or near the surface of the solution
- The molecule must have higher than average kinetic energy to overcome I_F

As shown in Figure 3.13.2, when an **aqueous solution** (water is the solvent of solution such as thin juice) absorbs heat, its molecules move faster, and water molecules at the surface eventually gain enough **kinetic energy**, K_E (energy due to the motion of the object) to escape the solution's surface and become the molecules of vapor. In other words, evaporation occurs when the E_K of the solution becomes greater than its I_F. (Note: Because we cannot see molecules' movements, we must imagine their changes to another state.)

If more heat energy is added to increase the temperature of the solution to its boiling point, the water molecules become more aggressive and start to produce bubbles, which rise to the surface, resulting in boiling and the release of vapor. As the molecules leave the liquid phase, they take their E_K with them, so vapor contains some energy. At the boiling point, the vapor pressure (↑) on the molecules is equal or greater than the total of atmospheric pressure (↓) and water pressure (→). (Arrows indicate the direction of forces.)

When a solution is heated, its vapor pressure increases until it equals the atmospheric pressure, at which point the solution begins to boil. In other words, the boiling point (*BP*) of

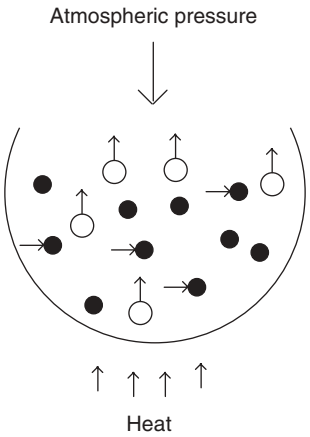


Figure 3.13.2 Evaporation at atmospheric pressure

a solution is the temperature at which the solution boils at atmospheric pressure (1 atm or 14.7 psi, the pressure at sea level).

EVAPORATION IN A SUGAR FACTORY

In sugar factories, evaporation conditions (explained in the preceding section) are easily met because evaporation is performed under low vacuum (flashing evaporation), which lowers the *BP* of the juice (*the lower the pressure, the lower is the BP*). The presence of a vacuum increases the temperature difference (ΔT) between the steam and the juice, so the juice boils at a lower temperature (*the higher the ΔT, the lower is the BP*). Energy is also saved because of the lower *BP* of the juice.

To obtain a low boiling temperature in the evaporators, a condenser keeps the juice under vacuum, usually with the help of a vacuum pump placed between the evaporator and the condenser. This means that the juice must be pumped out of the evaporators. The presence of the vacuum makes the juice boil at a lower temperature.

The steam pressure to the first effect is 240 to 380 kPa (35 to 55 psi). The final effect operates at near atmospheric pressure. The concentrator uses vapor from the second-effect or third-effect evaporator and operates at a slightly negative pressure (about 70 kPa or 10 psi). The low-raw (C) crystallizer (for the C product) uses vapor from the final-effect evaporator and operates at a vapor pressure of about 20 kPa (3 psi).

As was mentioned previously, the functions of the evaporation station in a sugar factory can be summarized as follows:

- Concentrates thin juice
- Produces condensate for boilers
- Produces vapor and condensate for heat and condensate users

The proper integration of these three functions results in efficient energy usage in the factory. (Steam production in the boiler house, its distribution, and its utilization in the **heat**

exchangers, **evaporators**, and **concentrators** are important to the energy efficiency of any sugar factory.)

Besides economical aspects, the color increase between thin juice and thick juice during evaporation is important. In normal beet processing and with SO_2 applied to the thin juice before entering the evaporators, a reasonable color rise (below 20%) between the thin juice and the thick juice is an indicator of proper evaporation operation.

In the evaporation station, thin juice with about 15% *DS* is heated in the thin-juice heaters to about 90°C. Then it is pumped into the evaporators to increase its concentration to about 60% *DS*. The steam gives up its heat to the juice and leaves the evaporators as vapor and condensate. The juice receives the heat, boils, loses water, and leaves the evaporators as thick juice. Vapor is supplied to vapor users, such as the crystallization station and heat exchangers. Condensate is mainly used as boiler feedwater and diffusion supply water in the factory. The heat in the condensate is also used to heat some in-process products such as diffusion juice.

NOTE

Sugar factories are not similar with respect to the operating conditions and equipment in their evaporation station. Factors differ particularly in the methods of vapor distribution by heat users in their functionalities. Therefore, the explanations in this section can be used only as a general guide.

MULTIPLE-EFFECT EVAPORATION

A multiple-effect evaporation station consists of usually three to six **effects**. Often, each effect consists of one to three evaporators, called **bodies**. For example, a five-effect evaporation station with two bodies in each effect contains 10 evaporators.

A **single-effect evaporation** operation is not efficient because the vapor has a large amount of energy and low pressure. Therefore, sugar factories use multiple-effect evaporation, in which the partially concentrated juice leaving the first effect is introduced into the second effect, and the vapor produced from the first effect is used as a heating medium to heat the second effect. After additional concentration, the product from the second effect becomes feed for the third effect, and so on. Vapor from the last effect goes to a condenser. Finally, the product from the last effect with the desired final concentration is pumped out of the last effect.

In multiple-effect evaporation, steam is used only in the first effect, which results in considerable energy savings for the factory. Each effect is equipped with a **condensate flash tank** that converts condensate to vapor by lowering the pressure to be used in the following effect. Flashing reduces steam consumption because the flash heat of each effect contributes to evaporation efficiency of the next effect.

A single-effect evaporation needs roughly 1 kg of steam to evaporate 1 kg of water in the product. But in a multiple-effect evaporation, 1 kg steam entering the first effect can evaporate as many kilograms of water as there are effects in the system. In other words, the steam is used as many times as there are effects in the system because 1 kg of steam entering the first effect evaporates 1 kg of water from the juice and produces about 1 kg of vapor. One kg of vapor from the first effect enters the second effect, where it evaporates 1 kg of water and produces 1 kg of vapor, which enters the third effect, and so on.

Beet-sugar factories usually use three to six effects, but four or five effects are the most common. Figure 3.13.3 shows a five-effect evaporation station, and Figure 3.13.4 shows the first effect of a five-effect evaporation station. Today, thick-juice concentrator or pan crystallizers are commonly used as the last effect of the evaporation station. This considerably increases the steam efficiency of the factory.

NOTE

Although a multiple-effect evaporation provides considerable steam saving compared to a single-effect evaporation, its total capacity is not greater than that of a single effect, given the same heating surface (McCabe et al. 2001).

Capacity and Economy of Multiple-Effect Evaporation

If the feed to the evaporator is at its boiling point, all the heat provided by the steam is available for evaporation, and the **capacity** is proportional to the rate of heat (Q). The **temperature difference** (temperature drop) between the heating medium and feed is the *driving force* for heat transfer and the capacity of the evaporation station. For example, thin juice can be heated to as high as 135°C in the first effect to increase the ΔT between the first and the last effect, which increases the capacity of the evaporation station and improves steam economy.

The temperature difference depends on the following:

- **Boiling point elevation** (*BPE*): The smaller the *BPE*, the larger is the ΔT and the better is the steam efficiency. In other words, if water is evaporated instead of juice in the evaporators, the ΔT will be greater because there is no *BPE*. Therefore, the *BPE* considerably affects the evaporation process when the *DS* of the juice increases (*the larger the DS, the larger is the BPE and the smaller is the* ΔT).

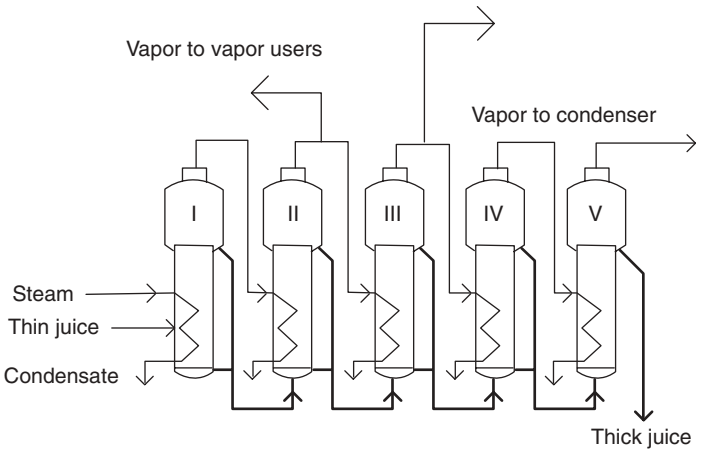


Figure 3.13.3 Five-effect evaporation station



Figure 3.13.4 First effect of a five-effect evaporation station

- **Liquid head** (juice level): The larger the liquid head (the juice level over the heating surface), the *higher is the pressure due to hydrostatic head, the smaller is the ΔT, and the lower is the steam efficiency*. When the pressure becomes higher, the **heat transfer** (U) becomes lower because the ΔT across the tube is lower, and the juice boils at a high temperature.

The **steam economy** (SE) of the evaporation station is the ratio of mass of water evaporated (M_W) per mass of steam used (M_S).

$$SE = \frac{M_W}{M_S} \tag{3.13.1}$$

The three main factors affecting the economy of an evaporation station are the following:

- Feed temperature
- Number of effects
- Multiple use of heat

The **feed temperature** (thin-juice temperature) entering the evaporation station is important because if the juice temperature is low, part of the enthalpy of the steam is used to increase its temperature to the boiling point and only the rest is used for evaporation.

The **number of effects** in the evaporation station affects steam economy considerably. A typical value for the economy of a single-effect evaporator is close to 1 and increases as the number of effects increases. In a single-effect evaporation, 100 kg steam are needed to evaporate 100 kg of water. In a two-effect, the steam requirement is 100/2 = 50 kg. In a three-effect, 100/3 = 33 kg is needed. In a four-effect evaporation, 25 kg, and in a five-effect evaporation, 20 kg. The steam savings from a single effect versus a two-effect is 100 − 50 = 50 kg. From a four-effect to a five-effect evaporators, however, the steam saving is only 5 kg (25 − 5 = 5), which is 10 times less. (Therefore, there is *a limit to the number of effects that work efficiently*.) In sugar factories, evaporation stations with four to five effects are the most common.

The optimum number of effects can be determined from an economic evaluation of the savings in steam gained by multiple-effect operation and the added investment required. Note: The investment needed for an *N*-effect evaporator is about *N* times that for a single-effect evaporator of the same capacity (McCabe et al. 2001).

Multiple use of heat refers to heating with vapor or condensate produced in individual effects instead of steam. The amount of heat required by **heat users** is about *30 to 40% of the steam usage* and is higher than that used by evaporators. Proper management of multiple-use of heat improves the energy economy of the evaporation station because heat in most heat users is subsequently returned to the evaporation station. For example, heat used to increase thin-juice temperature is returned to the evaporation station in thin juice.

Following are some heat users:

- Diffuser
- White pan
- Low-raw pan
- High-raw pan
- Raw-sugar melter
- Thin-juice heaters
- Diffusion-juice heaters
- Second-carb juice heaters
- Standard-liquor concentrator

In a five-effect evaporation using exhaust steam, vapor from different effects can be distributed as follows:

- First vapor can be used for thin-juice heaters
- Second vapor can be used for thin-juice heaters
- Third vapor can be used in white and low-raw pan crystallizers
- Fourth vapor can be used in prelimed juice heaters, standard liquor, and high-raw pan
- Fifth vapor can be used in diffusion-juice heaters and some heaters from carbonation station

Most important is the type of vapor used in the crystallization station. Multiple-effect evaporation not only reduces the amount of required steam for evaporators but also produces vapor that can be shared by heat users.

The amount of **steam reduction** (*SR*, in kg, tons, or % OB) achieved by moving the vapor to a lower-grade vapor (bleed) can be expressed by a simple equation called the **vapor-shift formula**:

$$SR = \frac{V_2 - V_1}{N} \tag{3.13.2}$$

N	Number of effects in the evaporation station
V_2	Number of effects from which the vapor was drawn after the change
V_1	Number of effects from which the vapor was drawn before the change

EXAMPLE 1

In a five-effect evaporator, what is the *SR* if the first or second vapor is used instead of exhaust steam (at 3 bar or 43.5 psi) to heat the thin-juice heater, which needs 8 kg steam?

When steam is shifted from exhaust steam to first vapor, the reduction is

$$SR = \frac{V_2 - V_1}{N} = \frac{1-0}{5} = \frac{1}{5} = 0.2\,\text{kg}$$

Because this heater needs 8 kg vapor, the vapor required after shifting to first vapor is

$$8 - 8 \times 0.2 = 6.4\,\text{kg}$$

When shifting to second vapor, the *SR* is

$$SR = \frac{V_2 - V_1}{N} = \frac{2-0}{5} = \frac{2}{5} = 0.4\,\text{kg}$$

$$8 - 8 \times 0.4 = 4.8\,\text{kg}$$

Other factors affecting the economy of the evaporation station are as follows:

- Diffusion draft
- Crystallization scheme
- Condensate distribution
- Thick-juice dry substance
- Capacity of the evaporation station
- Amount of backboiling in the sugar-end
- Amount of wash water used on the filters
- Amount of wash water used on the centrifuges
- Amount of evaporation required in the white pan crystallizers

HEAT EXCHANGERS

In the sugar factory, **heat exchangers** (heaters) are used to heat in-process products (juices, syrups, and liquors). They are important to the steam economy of the factory because in-process

products are usually heated for better performance. For example, to decrease the temperature difference between the juice and the steam entering the evaporation station, the juice is heated in thin-juice heaters to a temperature close to boiling. The heat exchangers are usually heated by using vapor or condensate supplied by the evaporation station. Two types of heat exchangers are used in sugar factories:

- Tube heat exchanger
- Plate heat exchanger

A **tube heat exchanger** (see Figure 3.13.5) consists of a pipe located inside another pipe. The vapor or condensate is introduced between the pipes, and the juice moves inside the smaller pipe.

A **plate heat exchanger** (see Figure 3.13.6) consists of a set of steel parallel plates. The plates are closely spaced and assembled between two frames, compressed by tightening bolts. The plates have a hole on the ends to direct the flow of the liquid and are placed so that the juice can circulate between them. Rubber gaskets seal the plates' edges and holes to prevent the intermixing of the juice and heating media. The gaskets may be a limiting factor, depending on the temperature of the heating medium (rubber gaskets cannot resist high temperatures). The direction of the feed and the heating flow can be concurrent or countercurrent. The arrangement of the holes on each plate directs the passage of the juice. Plates are pressed (corrugated) in a way that creates a special pattern to improve the feed movement, the heat transfer, and mechanical strength. In some heat exchangers, the plates are made of stainless steel.

The plate heat exchangers can handle a high flow rate (up to 20 t per hour). They are becoming popular in modern sugar factories because of the following advantages:

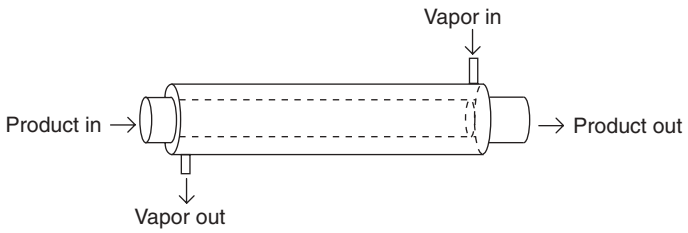


Figure 3.13.5 Tube heat exchanger

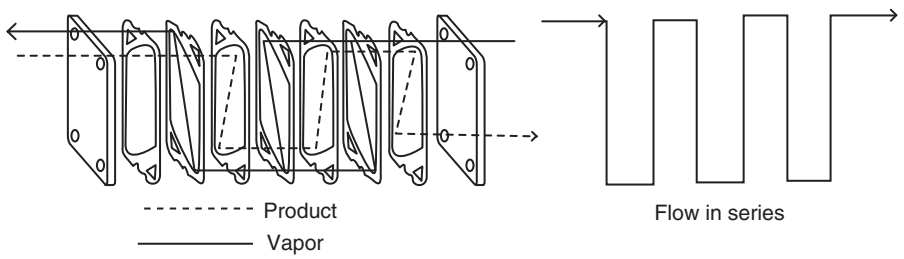


Figure 3.13.6 Plate heat exchanger

- Low floor area
- Simple maintenance
- Low capital investment
- Capacity can be increased by adding more plates to the frame
- Operate satisfactorily, particularly for heating low-viscosity products

The disadvantage of these heaters is the fast buildup of scale deposition on the surface of the plates due to the close spacing between plates. Scale deposition decreases the heat transfer rate, so the plates should be cleaned at regular intervals.

TYPES OF EVAPORATORS

In general, each evaporator consists of a vessel and heating tubes. The vessel is usually constructed from iron sheets, and the heating tubes are made of iron or stainless steel. Evaporators can be divided according to the shape of their heat-transfer surface:

- Tube evaporators
- Plate evaporators

Tube evaporators are further divided into the following subgroups:

- Robert evaporators
- Thin-film evaporators (rising-film and falling-film evaporators)

Sugar factories may use more than one type of evaporators.

Robert Evaporators

Robert evaporators (Figure 3.13.7) are the most used evaporators in sugar factories and are named after the inventor. A Robert evaporator consists of a calandria located in the bottom of the evaporator. In Robert evaporators, the heating tubes are relatively shorter (2 to 3 m) compared with those in thin-film evaporators (5 to 10 m).

The **calandria** is the heating section and the steam chest of an evaporator. It consists of several tubes and two perforated sheets, called **tube sheets**. The tube sheets are placed horizontally at a distance of 2 to 3 m apart. The bottom sheet is placed slightly above the base of the evaporator. The holes in the tube sheets are the size of the heating tubes (30 to 35 mm in diameter). The heating tubes are rolled into the holes of the tube sheets. A wider tube (400 to 600 mm in diameter), called the **downtake tube** (circulation tube), is welded in the center of the steam chest to the tube sheets.

A separator at the top of the evaporator removes juice droplets carried by the vapor. The evaporator usually has three **sight glasses**, placed one above the other, above the steam chest for observing the juice level in the evaporator. The evaporator also has a thermometer and a pressure gauge.

Steam is introduced into the steam chest between the tubes. The boiling juice is inside the tubes, below and above the tube sheets, without direct contact with the steam. The juice flows downward in the downtake tube to the bottom section, and then moves upward in the heating tubes. The temperature difference between the juice and the steam causes boiling. Heat from the steam passes through the tubes to the juice, evaporating water in a quantity equal to

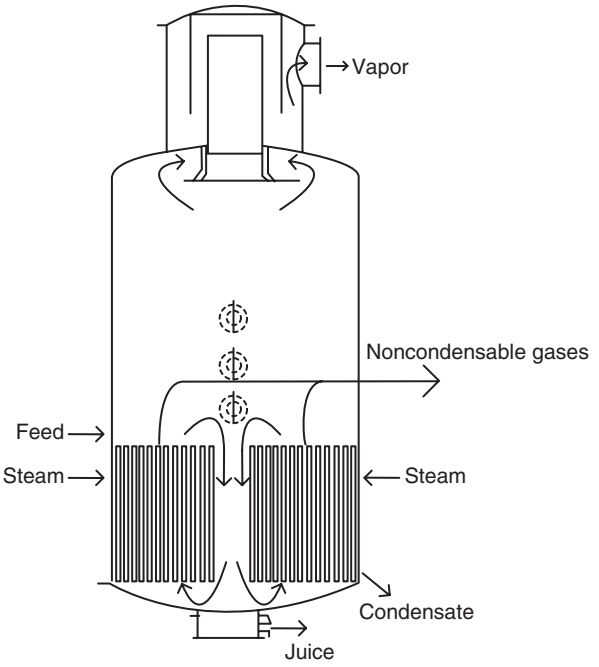


Figure 3.13.7 Robert evaporator

the mass of the steam condensed. Vapor formed from the boiling juice rises to the top and leaves the evaporator. The steam gives up heat and begins to condense. The condensate is pumped out from the base of the steam chest. The condensing process creates a low vacuum in the evaporator.

Noncondensable gasses (e.g., air, ammonia, and carbon dioxide gas) are discharged from the base of the steam chest. In a **multiple-effect evaporation** (explained later), steam in the first-effect chest usually carries a small amount of noncondensable gasses, so it is vented to the atmosphere. The vent from the second-effect steam chest receives most of the noncondensable gasses. This is one of the reasons for supplying boiler feedwater mainly from the condensate produced in the second-effect evaporators.

Thin-Film Tube Evaporators

The thin-film tube evaporator (TFT evaporator) has been used in some sugar factories since the 1980s. In a TFT evaporator, a thin layer (film) of feed like that of a soup bubble, is called **thin-liquid film**, and moves on the surface of the heating tubes. The layer's thickness is in the millimeter range. The heating tubes to which steam is entered are 5 to 10 m long.

Following are the differences between TFT and Robert evaporators:

- **Movement of liquid and steam:** In TFT evaporators, liquid moves outside the heating tubes and steam inside. In Robert evaporators, the movements are reversed.

- **Inventory:** In TFT evaporators, the inventory of the boiling material is too small. In Robert evaporators, the inventory is relatively large. (Small inventory can reach the desired concentration in a single pass, which is an advantage.)
- **Retention time:** The retention time is lower (1 to 4 minutes) in TFT evaporators than in Robert evaporators (several minutes), so less *color increase* occurs in thin-film evaporators (*color formation is proportional to retention time*). During evaporation, color intensity is due more to retention time than to any temperature increase.
- **Boiling-point elevation** (*BPE*): Unlike the Robert evaporators, there is no *BPE* in TFT evaporators because of the short retention time (the smaller the *BPE*, the larger is the ΔT and the better is the steam efficiency).
- **Heat-transfer coefficient** (*U*): *U* is higher in TFT evaporators than in Robert evaporators, so the **steam efficiency** is better in TFT evaporators.

In general, the limited range of concentration in TFT evaporators can be considered a big disadvantage. However, the boiling material can be recirculated to obtain a higher concentrated product. The two types of TFT evaporators are:

- Falling-film tube evaporator (FFT evaporator)
- Rising-film tube evaporator (RFT evaporator)

Falling-film tube evaporators (FFT evaporators) are used in modern sugar factories mainly as the final effect and concentrator, and in some factories as second to fifth effects. An FFT evaporator has round heating tubes 30 to 38 mm (1.25 to 1.5 in.) in diameter and 8 to 10 m (26 to 32 ft) long. A FFT 4 m in diameter has about 6000 m^2 (64 600 ft^2) of heating surface.

In an FFT evaporator, shown in Figure 3.13.8, a pump moves the juice to the upper surface of calendria. The distributor consists of perforated plates above a leveled tube sheet. The design of the distributors is complicated by the fact that every array must receive an equal share of juice. From the top, the juice uniformly falls by gravity through a distributor as a film and through the tubes at the velocity of 0.1 to 0.2 m/s (0.3 to 0.6 ft/s). The vapor also moves downward in the heating tubes and exits at the bottom of the evaporator. The two types of FFT evaporators are vertical tube and horizontal tube.

The advantages of FFT evaporators over RFT evaporators are the following:

- **Temperature difference:** The FFT evaporator can satisfactorily operate at much lower temperature differences than an RFT, allowing a larger number of effects to be incorporated. For example, suppose that the steam has a temperature of 130°C and the boiling temperature in the last effect was 70°C. Only four [(130 − 70)/14 = 4] effects are achievable in the RFT evaporator because it needs a 14°C temperature difference. But as many as 8 effects can be operated using FFT evaporators (an RFT evaporator needs a ΔT of only 6°C between the boiling liquid and the heating medium to operate well).
- **Viscosity:** The FFT evaporator can handle more viscous feed than the RFT evaporator.
- **Retention time:** The FFT evaporator has lower retention time (typically 1 minute), compared with the RFT evaporator (typically 4 minutes).
- **Feed movement:** Feed moves over the heating surface by gravity in an FFT evaporator but must be pumped in an RFT evaporator.

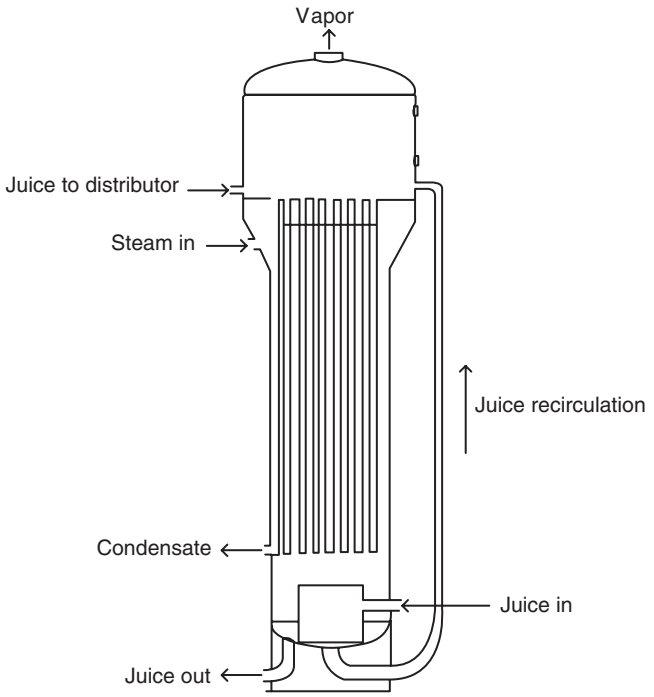


Figure 3.13.8 Falling-film tube evaporator

The **rising-film tube evaporator** (RFT evaporator) consists of long vertical heating tubes 5 to 10 m in height. The juice fed into the bottom of the evaporator moves upward over the tubes as a thin layer. (In the RFT evaporator, a temperature difference of at least 14°C between the boiling liquid and the heating medium is necessary to maintain a well-developed film.)

Plate Evaporators

Plate evaporators are the newest type and are the most energy-efficient evaporators. In these evaporators, the heating element consists of **plates** instead of tubes. Plates are 1.0 to 2.2 m (3 to 7 ft) high, and the distance between the plates is 6.0 to 7.4 mm. The plate evaporators are designed for evaporation duties using the advantages of Robert and thin-film evaporators and for correcting the disadvantages of plate heat exchangers. Plate evaporators:

- Have high *U*, similar to thin-film evaporators
- Have short retention time, similar to thin-film evaporators
- Operate in multiple-pass mode, similar to Robert evaporators
- Consist of plates, similar to plate heat exchangers, but the plates are welded

Plate evaporators have a very small feed volume, resulting in a short retention time (1 to 2 minutes compared to about 4 minutes in falling-film evaporators and several minutes in Robert evaporators). Plates are distributed in units (chambers) to which vapor is directed between the

plates. The juice boils on the surface of the plates as a film, allowing good heat transfer and a short retention time of the juice in the evaporator.

Plate evaporators offer the following benefits over Robert and thin-film evaporators:

- Require less energy
- Require less cleaning
- Take up less floor area
- Result in less color increase

The plate evaporators are of two types:

- Falling-film plate evaporator (FFP evaporator)
- Rising-film plate evaporator (RFP evaporator)

Since the early 1990s, beet-sugar and cane-sugar industries have installed several units of FFP evaporators made by different plate evaporator suppliers, such as Alfa Laval and GEA Ecoflex (see Figure 3.13.9). A new eight-effect evaporation station in Europe consists of six FFP evaporators (6500 m^2 each) and two falling-film tube evaporators. These evaporators have operated satisfactorily (Niepoth et al. 2005).

Today, some sugar factories are modifying their existing Robert evaporators to plate evaporators. In this retrofitting process, the existing Robert vessel stays in place without reconstruction, and the heating tubes are replaced with the plates, which increase the heating surface

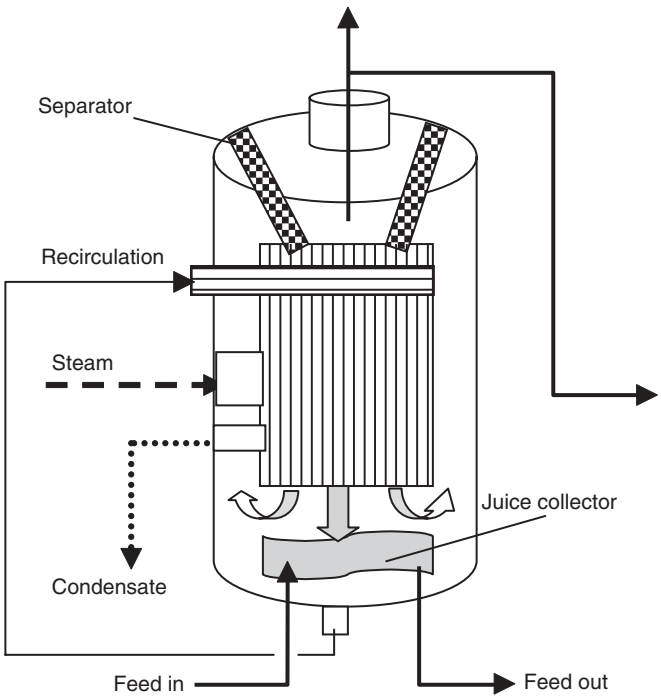


Figure 3.13.9 GEA plate evaporator

up to 300%. A retrofitted plate evaporator with 6000 m^2 of heating surface for use in a sugar plant has been reported (van der Poel et al. 1998).

CONCENTRATORS

Concentrators are used in sugar factories for concentrating thick juice or standard liquor to a definite *DS* content before feeding it to the crystallization station. Concentrators are helpful in the crystallization operation and the energy requirement of the factory because they evaporate part of water that otherwise must be evaporated in the pan crystallizers. A factory with a concentrator can concentrate the standard liquor to 70 to 75% *DS*, so that the liquor is already pre-evaporated before it reaches the pan crystallizers. This reduces the crystallization time of each strike in the first stage of crystallization by about half an hour.

The concentrator usually uses vapor from the second- or third-effect evaporators. Therefore, it can be considered as the last effect of the evaporation station, with the pan crystallizers being the following effects (pans also use the second or third vapor). Most concentrators are thin-film or plate-type evaporators.

EVAPORATION CONTROL AND ENERGY SAVING

In **multiple-effects evaporation**, the **temperature** and **pressure** of each effect is lower than that in the preceding effect. In a stable operation, the feed flow rate, temperature, and concentration are stable. The steam and condenser pressure and juice level in each effect are maintained. Therefore, changing the feed rate relative to the steam flow can change the concentration of thick juice (the juice leaving the evaporation station). If the thick juice is not thick enough, the feed rate to the first effect is reduced, and if the thick juice is too thick, the feed rate is increased. This results in a thick juice that eventually leaves the last effect with the desired concentration.

Following are some recommendations to lower the energy demands of the factory:

- Operate the factory at as steady a rate as possible.
- Use waste heat to heat the cold streams to about 60°C.
- Use the lowest effect vapors possible for crystallization.
- Use heat exchangers to cool hot streams instead of cooling water.
- Keep the evaporator's tubes **clean** by boilout for better heat transfer.
- Use condensate to heat some juice heaters and other possible vapor users.
- Never use **live steam** for anything other than the turbines and the evaporation station.
- Heat thin juice in the heaters (before entering the evaporation station) to the temperature slightly below its *BP*. This causes all the heat to be used in the evaporators for evaporation instead of heating the feed.
- Concentrate thick juice to a high dry substance (about 65%) to decrease the load of the crystallization operation. This is because the evaporation station acts as a multiple-effect operation, but each pan operates individually as a single effect.
- Remove **condensate** and **noncondensable gases** from the evaporators quickly and properly. Ammonia (a noncondensable gas) is easily removed because it acts as a **free** component rather than a combined component under alkaline conditions (see the following Notes).

- Keep the evaporator level at an optimum level because the efficiency of Robert evaporators largely depends on the juice level. Keeping a high juice level in the evaporator causes juice bubbles to rise to the top and enter the steam chest of the next effect evaporator. As a result sugar will be in the condensate, which is used as boiler feedwater. The sugar will deposit in the boiler heating tubes and cause considerable damage to the boiler operations.
- Keep the amount of excess vapor from the last effect at a minimum because the last vapor ends up in the condenser, wasting energy.

In modern sugar factories, digital and computer systems control the evaporator station. It is recommended that the evaporation station be *computerized* in such a way that its stability is not disturbed when other vapor and condensate users encounter problems. Controlling the evaporator **actuators** by computer and in a timely manner has the following benefits:

- Improves steam efficiency of the factory
- Controls the dry substance of thick juice by ±1%
- Controls the retention time in individual effects to lower color formation

The evaporation station is usually programmed based on the input and output values of the process, such as mass and energy balance equations and experimental knowledge.

NOTES

- **Running the evaporation station efficiently is one of the best ways to reduce costs in today's sugar factories, especially with the recent surge in fuel costs. In the United States, the steam consumption of the factories has recently been decreased to 25 and 28% OB. In newly built European factories, steam consumption on beet has been as efficient as 10%.**
- **When ammonia, which is alkaline, is removed during evaporation, the juice becomes less alkaline, and the pH decreases. This is known as a pH drop during evaporation.**
- **During evaporation, juice creates foam. When the amount of foam is high, it can cause entrainment (carrying juice along in an evaporator). To control foam, a defoamer is used.**

STEAM-PRESSURE REDUCTION AND VAPOR COMPRESSION

Steam-pressure reduction is the reduction of the pressure of the supersaturated steam by passing it through a **vapor-pressure reducer** before it enters the evaporator station**. Supersaturated steam** (all traces of water have been evaporated, and the steam acts as a pure steam) has lower heat transferability than **saturated steam** (some water still exists with the steam). Steam-pressure reduction automatically maintains the steam pressure at the required level, which is not affected by fluctuating steam consumption.

Vapor compression is the recompression of a lower value vapor by a **compressor** or a **steam-jet booster** (jet compressor). It is an action opposite to pressure reduction. To increase the heat value of a vapor exiting an individual effect of the evaporator station, the vapor is recompressed. The recompression increases vapor pressure and temperature, so the compressed vapor can be reused to evaporate more water. Vapor recompression improves the heat economy

of the factory (*the greater the heat economy in other stations, the more is the need for vapor compression*). Compression is usually applied to the first vapor, or the first and second vapor of the multiple-effect evaporation station.

The vapor-pressure compressors can be driven by an electric motor or a steam turbine, while the jet boosters are steam driven. Electrically driven compressors require high electricity, which affects the power consumption of the factory.

SCALE FORMATION IN EVAPORATORS

The common minerals present in thin juice are the following:

- Limesalts (hardness)
- Sodium compounds
- Silica dioxide (SiO_2)

Limesalts (dissolved Ca and Mg salts, also known as **hardness**) are the most scale forming of the minerals. Limesalts settle in the form of scale in the evaporators' tubes because of decrease in their solubility at higher concentrations. The heat coefficient then steadily decreases until the evaporator must be shut down to clean its tubes, known as evaporator **boilout** (cleaning). The rate of precipitation of limesalts is different in individual effects of a multiple-effect station (maximum precipitation occurs in 4^{th} and 5^{th} effects).

In terms of evaporator scaling, some types of hardness are harmful and some are less harmful. For example, hardness in thin juice in the amount of 300 mg/100 *DS* in one sugarbeet growing area can create a need for a lot of cleanings, while the same amount in other areas will not need as many cleanings. This depends on the type of hardness in the juice. For example, the hardness of Michigan sugar-beet factories belongs to the less harmful type, although the quantity is high (200 to 600 is not unusual). This amount in other areas, however, might necessitate many evaporator cleanings.

The precipitation and scaling of Ca, Mg, and Na compounds depends on temperature. But the precipitation of silica (Si) compounds is a matter temperature and pH of the solution (*the higher the pH, the lower is the rate of precipitation of Si*). Generally, the rate of precipitation of limesalts depends on their solubility (*the higher the concentration of the juice*, *the lower the solubility of limesalts, and the higher is the rate of their precipitation*).

The solubility of limesalts decreases (the rate of precipitation increases) when

- Alkalinity increases
- Temperature increases
- Amount of CO_2 gas decreases

As alkalinity increases, the solubility of limesalts decreases, so calcium and magnesium chloride and sulfate precipitate as $CaCO_3$ and $Mg(OH)_2$. But, as the alkalinity increases, the solubility of silica also increases, which prevents its precipitation (advantage).

During evaporation, part of the limesalts precipitate, forming *scale deposition* on the evaporator tubes. The rest passes the evaporation and crystallization process and ends up in molasses. The molasses produced in a factory with a juice-softening process is known as **hard molasses** (the molasses with the hardness content more than 3 meq CaO/100 *DS*).

NOTES

- **The *rate of precipitation* of hardness in the evaporation process is important to sugar processing because it affects the cleaning interval of the evaporators.**
- **The alkalinity and pH drop during evaporation is usual and is caused by the decomposition of amides and invert sugar and the escape of ammonia at high temperatures. A pH drop of 0.2 to 0.4 during evaporation is usual; anything above 0.4 is considered unusual. The alkalinity and pH drop also continues in the crystallization station.**
- **The amount of deposit (scale) on the heating tubes can be judged by the increase in temperature difference between the different effects. For example, if the usual temperature difference between the second and third effect is 10°C, any increase in difference (e.g., to 12°C) is a sign of scaling in third-effect tubes and indicates that it needs cleaning.**
- **In the evaporation operation, scaling decreases the heat transfer rate from the heating medium to the juice, causing a gradual drop in the pressure drop in individual effects.**

EVAPORATOR CLEANING

Evaporator cleaning (boilout) refers to using chemicals to remove scale (deposits) from the evaporator tubes. An increase in the temperature difference and the increase in pressure drop between individual effects of the evaporators are indications of dirtiness in an individual effect (see the Operating Problems at the end of this section for numerical data). The laboratory should keep a logbook to record the temperature and pressure in individual effects of the evaporation station.

Usually, the cleaning is performed without shutting down the evaporation station by bypassing the effect that has to be cleaned. You can clean a scaled evaporator according to the following procedure:

1. Drain the evaporator and rinse it with condensate.
2. Fill it with 4% soda ash (Na_2CO_3) and 1% caustic (NaOH) solutions.
3. Boil the chemicals for 5 to 10 hours to turn the calcium sulfate and oxalate into $CaCO_3$:

$$CaSO_4 + Na_2CO_3 \rightarrow CaCO_3 + Na_2SO_4$$

$$CaC_2O_4 + Na_2CO_3 \rightarrow CaCO_3 + Na_2C_2O_3$$

4. Drain the evaporator from the chemicals and fill it with 3 to 5% hydrochloric acid containing an *inhibitor* to protect the metal of the evaporation equipment against *corrosion*. In the case of stainless steel, **formic acid** (HCOOH) or sulfamic acid is used.
5. Heat the evaporator to about 70°C for about 3 hours to decompose the calcium carbonate:

$$CaCO_3 + 2HCl \rightarrow CaCl_2 + CO_2 + H_2O$$

6. Drain the acid solution from the evaporator and fill it with 1% soda ash (Na_2CO_3).
7. Rinse the evaporator again with condensate.

MATERIAL AND HEAT BALANCE

The economy of the evaporation station is a matter of material and particularly energy (heat) balance. The data provided by the instruments of the evaporation station can be used to

calculate material and energy balance. Following are some of the parameters under the control:

- **Flow:** Thin juice and thick juice.
- **Pressure:** Steam input and vapor leaving the station.
- **Density for indication of concentration:** Juice between evaporator effects and the thick juice.
- **Temperature:** Steam input, vapor output, juice flow, juice between evaporators, juices in recirculation, and condensate.

Material Balance

Assuming no loss, the material balance of an evaporation system can be expressed by the following mass equation:

$$M_W = M_V = M_F - M_P \tag{3.13.3}$$

M_W	Mass of evaporated water
M_F	Mass of feed (thin juice)
M_P	Mass of product (thick juice)
M_V	Mass of vapor

Masses are in kg, t, lb, or % on beet (OB). The mass (amount) of product will be less than feed by that many times as the concentration has increased:

$$M_P = M_F \times \frac{DS_F}{DS_P} \tag{3.13.4}$$

DS_F	Dry substance of feed
DS_P	Dry substance of product

You can calculate the amount of feed (thin juice) to the evaporator with the following equation:

$$M_{\text{Thin J.}} = M_B \times \frac{D}{100} \times \frac{DS_{\text{Dif. J.}}}{DS_{\text{Thin J.}}} \tag{3.13.5}$$

D	Draft (mass of diffusion juice % OB)
M_B	Mass of beet processed

The amount of thick juice is

$$M_{\text{Thick J.}} = M_{\text{Thin j.}} \times \frac{DS_{\text{Thin J.}}}{DS_{\text{Thick J.}}} \tag{3.13.6}$$

The amount of evaporated water (M_W) can be calculated as follows:

$$M_W = M_{\text{Thin J.}} - M_{\text{Thin J.}} \times \frac{DS_{\text{Thin J.}}}{DS_{\text{Thick J.}}}$$

$$M_W = M_{\text{Thin J.}} \left(1 - \frac{DS_{\text{Thin J.}}}{DS_{\text{Thick J.}}}\right) \tag{3.13.7}$$

The M_W is found from the difference of $M_{\text{Thin J.}}$ and $M_{\text{Thick J.}}$:

$$M_W = M_{\text{Thin J.}} - M_{\text{Thick J.}} \tag{3.13.8}$$

For calculating the amount of evaporated water in each effect of a five-effect evaporation station, assume that x kg is the amount of water evaporated in the fifth effect:

$$M_{W5} = x\,\text{kg}$$

This means that there is a need of x kg of fourth-effect vapor for evaporating x kg of water from the fifth effect:

$$M_{W4} = M_{V4} = M_{V5} = x\,\text{kg}$$

In a five-effect evaporator, the general equation can be expressed as follows:

First effect	$x + M_{V1} + M_{V2} + M_{V3} + M_{V4} = M_{W1}$
Second effect	$x + M_{V2} + M_{V3} + M_{V4} = M_{W2}$
Third effect	$x + M_{V3} + M_{V4} = M_{W3}$
Fourth effect	$x + M_{V4} = M_{W4}$
Fifth effect	$x = M_{W5}$
Total	$5x + M_{V1} + 2M_{V2} + 3M_{V3} + 4M_{V4} = M_{W5}$

$$x = M_{W5} = \frac{M_W - (M_{V1} + 2M_{V2} + 3M_{V3} + 4M_{V4})}{5} \tag{3.13.9}$$

Therefore, the **total steam requirement** (M_S), which is used in the first effect, is

$$M_S = M_{S1} = \frac{M_{W5} - (M_{V1} + 2M_{V2} + 3M_{V3} + 4M_{V4})}{5} + M_{V1} + M_{V2} + M_{V3} + M_{V4} \tag{3.13.10}$$

This value is almost the same as the steam entering the first effect, less the amount of steam required to raise the thin-juice temperature to the boiling point.

NOTE

For better steam economy, the amount of the excess vapor from the last effect should be minimal because this vapor ends up in the condenser and is therefore lost (as waste heat to the cooling water). As seen in Eq. (3.13.10), lowering the amount of vapor from the last effect can be achieved by decreasing the total amount of water to be evaporated (M_W), by increasing the sum of ($M_{V1} + 2M_{V2} + 3M_{V3} + 4M_{V4}$), or by using that vapor to do meaningful heating in the factory. To decrease the M_W, the diffusion draft (D) should be reduced. Decreasing the draft can work to some extent because the amount of vapor produced by the evaporators for heat users is also reduced, so there is a need for additional live steam, which is not economical. To increase the ($M_{V1} + 2M_{V2} + 3M_{V3} + 4M_{V4}$), the amount of additional vapor for use by other heat users should be increased. This is the best solution because, as mentioned, heating with vapor instead of live or exhaust steam (low-grade vapor utilization) improves steam economy.

EXAMPLE 2

Given:

A 5-effect evaporation station	
Draft (D)	120.0%
Dry substance of diffusion juice ($DS_{\text{Dif. J}}$)	16.0%
Dry substance of thin juice ($DS_{\text{Thin J}}$)	15.0%
Dry substance of thick juice ($DS_{\text{Thick J}}$)	65.0%

Calculate:

- Amount of thin juice
- Amount of thick juice
- Amount of evaporated water
- Amount of evaporated water in each effect in a five-effect station when there is a need for **additional vapor** (AV) for other heat users such as: $M_{AV1} = 15$ kg from the first effect, $M_{AV2} = 10$ kg from the second effect, and $M_{AV3} = 5$ kg from the third effect.
- Steam economy (steam consumption % OB)

Draft of 120% means that 120 kg (tons or pounds) of juice is produced from 100 kg (tons or pounds) of sugarbeet. This means that the following calculations are based on 100 kg beet (% OB).

The amount of thin juice can be calculated from Eq. (3.13.5):

$$M_{\text{Thin J.}} = M_{\text{Beet}} \times \frac{D}{100} \times \frac{DS_{\text{Dif. J.}}}{DS_{\text{Thin J.}}} = 100 \times \frac{120}{100} \times \frac{16.0}{15.0} = 128 \text{ kg or \% OB}$$

The amount of thick juice is calculated from Eq. (3.13.6) as

$$M_{\text{Thick J}} = M_{\text{Thin J}} \times \frac{DS_{\text{Thin J}}}{DS_{\text{Thick J}}} = 128 \times \frac{15.0}{65.0} = 29.5$$

The amount of evaporated water can be found from Eq. (3.13.8) as

$$M_W = M_{\text{Thin J.}} - M_{\text{Thick J.}} = 128 - 29.5 = 98.5$$

This means that the evaporation station should evaporate 98.5 kg water for each 100 kg of sugarbeet processed. The total of the mass of evaporated water (or produced vapor) in all effects is equal to $M_W = 98.5$ kg (or % on beet, OB)

$$M_W = M_{W1} + M_{W2} + M_{W3} + M_{W4} + M_{W5} = 98.5$$

or

$$M_V = M_{V1} + M_{V2} + M_{V3} + M_{V4} + M_{V5} = 98.5$$

Assume that the total mass of water evaporated in the fifth effect is equal to x

$$M_{W5} = M_{V5} = M_{W4} = x \text{ kg}$$

In the fourth effect, x kg of vapor is needed for evaporating x kg of water from the fifth effect, so the total mass of water evaporated in the fourth effect is

$$M_{W4} = M_{V4} = M_{W5} = x\,\text{kg}$$

In the third effect, x kg of vapor is needed for evaporating the x kg of water from the fourth effect. In addition to x kg of vapor directed to the fifth effect, $M_{EV4} = 5$ kg of vapor was used in heat users. So $x + 5$ kg water must be evaporated. Therefore, the total mass of water evaporated in the third effect is

$$M_{W3} = M_{V3} = M_{W4} = x + 5\,\text{kg}$$

Similarly for the second effect from which 10 kg of additional vapor was drawn for heat users,

$$M_{W2} = M_{V2} = M_{W3} = x + 5 + 10$$

The evaporated water in the first effect is

$$M_{W1} = M_{V1} = M_{W2} = x + 5 + 10 + 15$$

The total mass of evaporated water (M_W) is 98.5 kg, so x can be calculated:

$$\begin{aligned} x = M_{W5} &= x + 5 + 10 + 15 + x + 5 + 10 + x + 5 + x + x = 98.5 \\ 5x &= 98.5 - 50 \\ x &= 9.7\,\text{kg} \end{aligned}$$

Now, the mass of water evaporated or the vapor produced in each effect can be calculated:

$$\begin{aligned} M_{W1} = M_{V1} &= x + 5 + 10 + 15 = 9.7 + 30 = 39.7 && \text{kg} \\ M_{W2} = M_{V2} &= x + 5 + 10 = 9.7 + 15 = 24.7 && \text{kg} \\ M_{W3} = M_{V3} &= x + 5 = 9.7 + 5 = 14.7 && \text{kg} \\ M_{W4} = M_{V4} &= 9.7 && \text{kg} \\ M_{W5} = M_{V5} &= 9.7 && \text{kg} \end{aligned}$$

$$M_W = M_V = 39.7 + 24.7 + 14.7 + 9.7 + 9.7 = 98.5 \quad \text{kg}$$

The **steam requirement** (M_S) can be calculated from the amount of water evaporated from the first effect. We already found that 39.7 kg water was evaporated from the first effect. Therefore, 39.7 kg of steam is required to process 100 kg of beet (see the following Note).

$$M_{W1} = M_S = 39.7\,\text{kg (or \% OB)}$$

The M_S can also be calculated from the total amount of water evaporated from the last effect and extra vapor (**EV**) drawn from individual effects

$$M_S = M_{W5} + M_{EV1} + M_{EV2} + M_{EV3} = 9.7 + 15 + 10 + 5 = 39.7\,\text{kg (or \% OB)}$$

The **steam economy** (*SE*) is obtained from Eq. (3.13.1):

$$\mathbf{SE} = \frac{M_W}{M_S} = \frac{98.5}{39.7} = 2.5$$

This means that for each kg of steam, 2.5 kg of water was evaporated.

NOTE

The actual steam requirement of efficient factories is considerably lower (about 25% OB)

Heat Balance

According to the **Law of Conservation of Energy**, heat (or any type of energy) cannot be destroyed but can only be transferred between systems. In evaporation, with the assumption of *no heat loss* and *no use of saturated steam*, the **heat** (energy) balance is

$$(\text{Heat})_F + (\text{Heat})_S = (\text{Heat})_V + (\text{Heat})_P + (\text{Heat})_C$$

F	Feed
S	Steam
V	Vapor
P	Product
C	Condensate

Heat is expressed as Joule (**J**, rhymes with tool), British thermal unit (Btu), or calorie (cal). An **enthalpy** (H, the heat content of a substance at constant pressure) balance conducted on the evaporator system gives the following expression:

$$M_F H_F + M_S H_S = M_V H_V + M_P H_P + M_S H_C \tag{3.13.11}$$

M	Mass flow rate
H	Enthalpy expressed in kJ/kg or Btu/lb (for conversion: 1 Btu/lb = 2.33 kJ/kg)

The first term in Eq. (3.13.11) represents the heat coming with the feed (Q_F, or heat content of the feed). Q_F can be calculated with the following equation:

$$Q_F = M_F H_F = M_F C_{PF} \Delta T = M_F C_{PF} T_F \tag{3.13.12}$$

Q_F	Heat in kJ, Btu (for conversion: 1 kJ = 1.05 Btu) or kcal (1 kcal = 4.184 kJ)
C_{PF}	Specific heat capacity of feed in kJ/kg.°C or Btu/lb.°F (for conversion: kJ/kg.°C/4.187 = Btu/lb.°F)
ΔT	Temperature difference; **delta** (Δ) means difference and T is for temperature (for conversion: °C × 1.8 + 32 = °F)
T_F	Temperature of feed

The term $M_P H_P$ in Eq. (3.13.11) is the enthalpy of the product (Q_p) leaving the evaporator. It can be calculated from the following equation:

$$Q_P = M_P H_P = M_P C_{PP} T_P \tag{3.13.13}$$

C_{PP}	Specific heat capacity of product
T_P	Temperature of product

Specific-heat capacity (C_P, often called simply **specific heat**) is expressed in kJ/kg.°C (reads kilojoules per kilograms per degrees Celsius). kJ/kg.°C is equal to kJ/kg.K because a 1° change in temperature is the same on the Celsius or the Kelvin scale. In sugar-factory practices, the specific heat of juice can be calculated by using the dry substance of the juice (DS_{Juice}):

$$C_{\text{Juice}} = 4.187(1 - 0.006 DS_{\text{Juice}})\,\text{kJ/kg.°C} \tag{3.13.14}$$

or

$$C_{\text{Juice}} = 1 - 0.006DS_{\text{Juice}} \text{ Btu/lb.°F} \tag{3.13.15}$$

In Eq. (3.13.11), H_S and H_V can be obtained from the steam table (Table A.10 in the Appendix)

The **heat transfer** ($\underline{Q}$, absorbed and relersed heat) through the heating area of an evaporator is

$$\underline{Q} = U.A.(T_S - T_V) = M_S H_V - M_S H_C \tag{3.13.16}$$

U Heat-transfer coefficient in kJ/m^2.°C or Btu/ft^2.°F, or W/m^2.°C (for conversion: kJ/m^2.°C / 5.678 = Btu/ft^2.°F and kJ/m^2.°C = W/m^2.°C)

A Heating area in m^2 or ft^2 (for conversion: m^2 × 10.76 = ft^2)

The U (see the Glossary for definition) is proportional to the boiling temperature of the juice (T) and improportional to the DS of the juice (*the lower the DS and the higher the T, the higher is the U*). The U for a typical Robert evaporator for a boiling juice with DS between 20 and 50% is 1000 to 3000 W/m^2.°C. **Swedish formula** can be used to calculate the U for practical purposes.

$$U = \frac{503T}{DS} \tag{3.13.17}$$

The difference between the enthalpy (the heat content) of the steam (H_S) and the enthalpy of the condensate (H_C) is called the **heat of condensation of steam** (λ_S). The heat that can be released by steam ($\underline{Q}_S$) can be calculated from the enthalpy balance.

$$\underline{Q}_S = M_S(H_S - H_C) = M_S\lambda_S \tag{3.13.18}$$

Heat of evaporation of feed (λ_F) is the heat required to evaporate a given quantity of the feed into vapor. Assuming no heat losses, the heat released from the steam equals the heat received by the feed ($\underline{Q}_S = \underline{Q}_F$). Therefore, numerically the heat of condensation and vaporization are the same with the opposite signs.

EXAMPLE 3

Given:

Feed flow rate to a single evaporator	100 kg/h
Feed dry substance F_{DS}	15.0%
Product dry substance (P_{DS})	60.0%
Feed temperature in	90°C
Product temperature out	115°C
Steam temperature in	130°C
Heat-transfer coefficient (U)	1400 kJ/m^2.°C (250 Btu/ft^2.°F or 1400 W/m^2.°C)
Heat of evaporation of water (λ_W) at 100°C	2257 kJ/kg

Calculate:

- Amount of steam required
- Steam required to evaporate 1 kg water
- Steam economy
- Required heating area (A)

The amount of product (thick juice) is calculated from Eq. (3.13.6):

$$M_P = M_F \times \frac{F_{DS}}{P_{DS}} = 100 \times \frac{15.0}{60.0} = 25\,\text{kg}$$

The amount of evaporated water can be found from Eq. (3.13.8):

$$M_W = M_V = 100 - M_P = 100 - 25 = 75\,\text{kg}$$

The specific heat of feed can be calculated from Eq. (3.13.14):

$$C_F = 4.187(1 - 0.006 \times 15.0) = 3.8\,\text{kJ/kg.°C}\ (0.9\,\text{Btu/lb.°F})$$

The specific heat of product can be calculated the same as the specific heat of feed:

$$C_P = 4.187(1 - 0.006 \times 60.0) = 2.7\,\text{kJ/kg.°C}\ (0.6\,\text{Btu/lb.°F})$$

The enthalpy (heat content) of feed is

$$H_F = 3.8(90 - 0) = 342\,\text{kJ/kg}$$

The enthalpy of product is

$$H_P = 2.7(115 - 0) = 311\,\text{kJ/kg}$$

The heat content of steam (H_S) and the heat content of condensate (H_C) at 130°C can be found from the steam table (Table A.10 in the Appendix):

$$H_S = 2720.5\,\text{kJ/kg}$$

$$H_C = 546.3\,\text{kJ/kg}$$

The enthalpy of vapor (H_V) at 100°C is 2676.1 kJ/kg (the boiling-point elevation of thin juice is 0.3 and negligible).

The amount of steam required can be calculated from Eq. (3.13.11):

$$M_F H_F + M_S H_S = M_V H_V + M_P H_P + M_S H_C$$

$$100 \times 342 + M_S \times 2720.5 = 75 \times 2676.1 + 25 \times 311 + M_S \times 546.3$$

$$M_S = 80.2\,\text{kg/h}$$

The steam required to evaporate 1 kg water is

$$\frac{M_S}{M_W} = \frac{80.2}{75} = 1.1\,\text{kg steam/kg water}$$

To calculate steam economy (see the following Note), Eq. (3.13.1) is used:

$$SE = \frac{M_W}{M_S} = \frac{75}{80.2} = 0.9$$

The heating area required (A) is calculated from Eq. (3.13.16):

$$A = \frac{M_S H_V - M_S H_C}{U(T_S - T_V)} = \frac{80.2 \times 2676.1 - 80.2 \times 546.3}{1420(130 - 100)} = 4.1\,\text{m}^2\,(44\,\text{ft}^2)$$

NOTE

In sugar-factory practices, the steam economy (*SE*) for an *N*-effect evaporator is calculated by the following simple equation:

$$SE = \frac{1.175}{N}$$

For example, for a five-effect evaporator:

$$M_S = \frac{1.175}{5} = 0.24\,\text{kg steam/kg water}$$

EXAMPLE 4

Given:

Exhaust steam pressure to an evaporator	415 kPa (60.1 psi)
First-vapor pressure	260 kPa (37.7 psi)
Amount of first-effect condensate (M_C)	20000 kg/h
Steam enthalpy (H_S) at 415 kPa (from steam table)	611 kJ/kg (262 Btu/lb)
Condensate enthalpy (H_C) at 270 kPa (from steam table)	546 kJ/kg (234 Btu/lb)
Heat of vaporixation of vapor (λ_V) at 270 kPa	2174 kJ/kg (933 Btu/lb)

Calculate:

The amount of vapor produced (MV) from flashing of first-effect condensate to the flash tank, to be used in the second effect

The heat released from condensate by flashing can be calculated from Eq. (3.13.17):

$$Q_C = M_C(H_S - H_C) = 20\,000(611 - 546) = 1300\,000\,\text{kg/h}$$

Amount of vapor produced is

$$Q_V = M_V \lambda_V \qquad M_V = \frac{1300000}{2174} = 598\,\text{kg/h}\,(1316\,\text{lb/h})$$

OPERATING PROBLEMS

As the heat center of the factory, **the evaporation station** is one of the most important stations in the sugar factory due to high-energy demand and large amounts of water that have to be evaporated from the juice. The evaporation station supplies the vapor to several stations in the factory, including the crystallization station. In addition, the evaporators provide condensate to the boilers and other process users. Any problem that delays the performance of the evaporator station will eventually affect the other stations as well. Therefore, the stability of the evaporation station must not be disturbed when other vapor and condensate users have problems. However, some operating problems in this station are unavoidable. Following are some of the problems you may encounter.

Low-Efficiency Evaporation

Following are several possible causes for low-efficiency evaporation:

- **Heating tubes of evaporators are not clean:** This is determined by checking to see whether there is an increase in the temperature difference between the individual effects. Unsatisfactory purification causes deposition of scale in the first effects of the evaporators (the presence of scale in the last effect is normal). Clean tubes maintain the heat-transfer coefficient (U) value at an optimum level and minimize the temperature difference between effects. A typical temperature difference between vapor and juice in a four-effect Robert type evaporation is as follows:

Effect	Vapor Temperature (°C)	Juice Temperature (°C)	Difference (°C)
First	125	120	5
Second	118	108	10
Third	107	95	12
Fourth	94	70	24

(Note: The temperature difference might be different in other types of evaporators.)

- **Wrong juice level:** The juice level over the heating surface (liquid head) in a Robert evaporator should be kept at such a height that the top tubes' sheet is just covered. A very low level reduces the efficiency of the evaporator, and may cause caramelization of sucrose, resulting in an increase in juice color. On the other hand, a high juice level decreases the temperature difference and heat transfer, which lowers evaporator efficiency.
- **Low juice temperature:** When the temperature of the juice entering the first effect is low, the amount of heat usage is increased because the juice has to be heated to boiling temperature before any evaporation can start.
- **Low thin-juice dry substance:** A high draft during the diffusion process might be the cause.
- **Low heat rate:** Steam supply from the boiler house might be low, or the vapor demand from the first effect for vapor users, such as crystallization, might be high.
- **Slow removal of condensate:** This happens when condensate removal from the steam chest is insufficient.
- **Slow removal of noncondensable gases:** This happens when removal (venting) of noncondensable gases is insufficient.
- **Insufficient heating surface:** When the heating surface of the evaporators is insufficient to concentrate the juice, try one or more of the following:

 Decrease the draft
 Increase the steam pressure
 Increase the temperature difference
 Increase the vacuum on the final effect

- **High amount of thin juice:** The factory is producing more juice than the evaporators can process.

- **Leaks in heating tubes:** If there is a leak in the heating tubes, vapor can get to the juice area and dilute the juice.
- **Wrong rate of vapor removal from individual effects**

High Sugar Loss during Evaporation

Possible causes of high sugar loss during evaporation are the following:

- **High foam content:** When foam covers the heating surface, **heat transfer** (*U*) decreases and retention time increases, which may increase sugar loss (due to the conversion to invert sugar) during evaporation. Damaged beets or unsatisfactory operation of the purification station is the cause for high foam content.
- **High juice concentration:** High juice concentration, particularly in the last effects, increases the viscosity of the juice and the retention time, so sugar loss is increased due to the conversion to invert sugar.

CHAPTER 3

SECTION 14

JUICE DECOLORIZATION AND SULFITATION

SUBSECTIONS

- Color Formation in Sugarbeet Processing
- Juice Decolorization
- Juice Sulfitation
- Operating Problems

ABOUT THIS SECTION **This section** explains **Station 14** (Juice Decolorization and Sulfitation) of sugarbeet processing. It answers three important questions:

- What are colorants
- How are colorants formed
- How can colorants be reduced

Coloring substances (colorants), their formation, and ways to prevent or reduce them in in-process products (juices, syrups, liquors, and massecuites) are important in sugar production because a high-color juice produces a high-color sugar, which is not desired.

Almost all sugar factories use sulfitation (adding SO_2 to the juice) to reduce color. In some factories, color is further reduced by an **ion-exchange resin** or **activated carbon**. The decolorization process is used for thick juice or standard liquor, as well as in the production of liquid sugar.

(Note: A considerable difference exists between decolorization and sulfitation.) The **decolorization process** (the treatment of the juice with ion-exchange resin or activated carbon) works as a **curative remedy**, meaning this process cures the high-color problem in the juice when the color already exists. The **sulfitation process** (adding SO_2 to the juice) functions as both a **curative** and **preventive remedy** because it reduces color and also prevents additional color formation in the next processing stations, where the temperature is high.

Beet-Sugar Handbook, by Mosen Asadi
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Beet-sugar factories with a good purification and crystallization station can produce low-color sugar with only juice sulfitation if they select an effective crystallization scheme and carefully monitor the process to prevent excessive color formation. However, some sugar factories use the decolorization process because of low-quality beets or insufficient capacity of the evaporation and crystallization stations, which increase color formation.

To familiarize yourself with color value in sugar processing, look at the following typical color range of a few in-process products, molasses, and granulated-refined (GR) sugar. Numbers are in IU (ICUMSA unit), measured at 420 nm. (Note: Because the color of in-process products is different from factory to factory and even in the same factory from one day to the next, these color ranges are approximate.)

Thin juice	1500 to 3000
Thick juice	2000 to 3500
Standard liquor	2000 to 3500
Molasses	40000 to 70000
Sugar	15 to 45

Coloring substances (colorants) causes darkness in food products as well as in sugar. They have a complex polymeric structure, composed of many organic compounds. In sugar factories, colorants are present in in-process products as part of nonsucroses (nonsugars). They are not dissolved in the juice but rather suspended because colorants belong to the **colloids** (high molecular substances, namely pectin, dextran, and coloring substances). Colloids are electro-negatively charged, so they can be absorbed by electro-positively charged substances such as active carbon or anion ion-exchange resin.

NOTE

The chemistry of color formation is too complex to indicate with simple chemical equations, so this section does not include the topic of color chemistry.

COLOR FORMATION IN SUGARBEET PROCESSING

Colorants (coloring substances) are not present in beet juice but are formed during the processing (*sugarbeet is an off-white color, but processed beet juice is colored*). During processing, colorants in in-process products form because of the following reasons:

- High pH
- High temperature
- Interaction of organic nonsugars

Temperature is the main factor in color formation. Generally, the colorants related to sugarbeet processing are formed under two temperature conditions, as follows:

- Colorants formed at low temperatures
- Colorants formed at high temperatures

Colorants Formed at Low Temperatures

The colorants formed at low temperatures are subdivided into two groups:

- **Colorants formed by oxidation reaction:** Oxidation reactions are the reaction of a substance with oxygen. Sugarbeet is off-white in color, and the juice inside the cells is colorless; however, juice produced by squeezing sliced beets (cossettes) turns to brown immediately when exposed to air. This indicates that at low temperatures, some organic **nonsugars** (nonsucrose substances) in the beet juice react with the oxygen in the air and produce coloring substances. For example, tyrosine (an amino acid) oxidizes easily to coloring and other substances at low temperatures. The same thing happens with cossettes when they are exposed to air; immediately after slicing, the surface of cossettes becomes gradually yellow and finally brown, but the inside of the cossettes stays unchanged.
- **Colorants formed by melanization reaction:** Melanization reactions are the interreaction of amino acids. Melanins, present in beet juice, are good examples of colorants formed by the melanization reaction. They are mostly formed in the interval between the slicing of the beet and the heating of the juice in the diffuser. Most of these colorants precipitate with lime and are removed during purification.

Colorants Formed at High Temperatures

The colorants formed at high temperatures are subdivided into two groups:

- **Colorants formed by the Maillard reaction: Maillard reactions** (browning reactions) are the reactions that occur between the reducing sugars (e.g., glucose and fructose) and amino acids at high temperatures. The formation of these colorants occurs through complex and multiple reactions. Melanoidins are good examples of colorants formed by the Maillard reaction. The Maillard reaction produces a high color and viscous juice, which reduces the crystallization rate (*high viscosity plays an opposing factor in the crystallization process*). The products of the Mailard reactions also have a particular smell and taste. Most of the colorants formed by the Maillard reaction are removed during purification, but because the high temperature is the *driving force* of their formation, they are again formed during evaporation and crystallization.
- **Colorants formed by the caramelization reaction: Caramel** is a brown-color, pleasant-tasting product formed from heating of sucrose (sugar) solutions. In terms of chemistry, caramel is the product of the caramelization reaction formed by the decomposition of sucrose to glucose, fructose, and finally to caramel at temperatures close to its melting point (the melting point of sucrose is about 185°C). This reaction is called the **caramelization of sugar**, which does not occur to an appreciable extent during normal operations of the sugar factory. Caramel is produced commercially by boiling fermentable sugars, such as sucrose and fructose, in the presence of ammonia. It is used in food products as a flavor and color enhancer.

The Maillard reactions are at their lowest activities when the juice is neutral or slightly acidic. Such a condition exists in the diffuser. The Maillard reactions are not active under alkaline and low temperature conditions (e.g., the purification process). But they become active under alkaline and high-temperature conditions (e.g., the evaporation and crystallization process).

The beet-sugar factories face high color of in-process products when they process damaged beets. A typical beet contains about 0.3% invert sugar (or about 0.5 to 1.2% on *DS* base), but a damaged beet contains about 2% on dry substance (*DS*). Sugar scientists found a linear correlation between the color of the juice and its invert-sugar content when damaged beets are processed. Figure 3.14.1 shows the relation between thick-juice color and invert-sugar content in beet.

So far, you found the answer to the first two questions, "What are colorants?" and "How are they formed?" Now, you will find the answer to the third question, "How can colorants be reduced?"

JUICE DECOLORIZATION

Juice decolorization is performed by two methods:

- Decolorization by ion-exchange resin
- Decolorization by activated carbon

Decolorization by Ion-Exchange Resin

Coloring substances present in in-process products can be removed by an **ion-exchange resin** (referred to from now on as resin). The resins (see Chapter 5 for more information) mostly used in decolorization are a **strong-base anion resin** (SBA resin) in chloride form (Cl^- form). Amberlite 900 and Dowex MSA-1 are examples of these types of resins.

Thick juice or standard liquor is usually used in the decolorization process by a resin. The concentration of the syrup is important because the feed load (bed volume/hour or BV/h) is chosen based on the concentration of the syrup. Usually, feed with suitable viscosity (juice with 55 to 65% *DS*) is fed to the decolorization columns. The feed temperature is kept at 80 to 90°C. When resin is exhausted (saturated) with color, it is regenerated with 10% NaCl. In modern systems, the regenerant is nanofiltered and the retentate is reused to minimize NaCl consumption.

The decolorization process generally consists of the following steps:

1. **Service:** Juice is passed continuously through the resin bed until the resin is nearly exhausted.

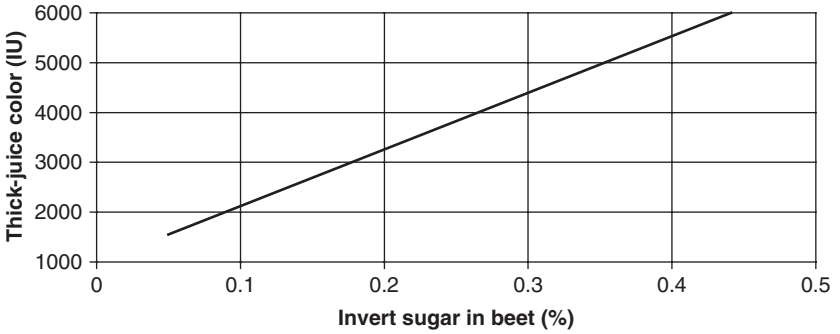


Figure 3.14.1 Relation between thick-juice color and invert-sugar content of beet

2. **Backwashing:** The resin is backwashed with soft water (condensate) to reclassify the resin particles. Backwashing brings the resin to its original order because during service, resin particles become classified (the largest particles remain at the bottom, while the smaller ones are distributed on a higher level). A backwash is provided by applying a uniform flow of water from the bottom of the bed to fluidize the resin bed. Typically, the amount of water required is about 2 bed volumes (BV).
3. **Regeneration:** The resin is regenerated with about 8% solution of common salt (NaCl) at about 10 BV (about 200 g salt per liter of resin). This returns the resin to its original form and capacity. It is recommended that the resin be regenerated with a lower concentrated salt solution (about 4%) every third or fourth cycle to increase its lifetime.
4. **Rinse:** The resin is rinsed with about 3 BV soft water.
5. **Service:** The regenerated column is then again fed with juice.

NOTE

In juice decolorization, polymeric decolorizing absorbents (e.g., Dowex Optipore or Ecosorb) can also be used because they have a high surface area and porosity (similar to activated carbon). Decolorizing absorbents are not used in beet-sugar industry because they cannot be regenerated (they do not have the functional group required for regeneration). This disadvantage increases the processing costs.

Decolorization by Activated Carbon

Activated carbon consists of particles of about 1 mm in diameter. It is produced from carbon-containing materials such as coal by high heat treatment (about 800°C) followed by a distillation process to remove the volatile components. The activated carbon is highly porous (so, it has a low density) with an extremely high surface area (about 1200 m^2/g) and is positively charged. Having a high surface area makes almost all ions of the activated carbon available for the absorption process. Activated carbon removes colorants from the juice based on the attraction of negatively charged colorants ions to the positively charged functional groups of the activated carbon.

Activated carbon can be used as a precoat in pressure-leaf filters at a dosage of 0.05 to 0.2% on solids of the juice. Usually, activated carbon is used to decolorize a low-color syrup, and then it is used for a higher-color juice. One or two passes are made, and then the filtered carbon is discarded. Activated carbon not only removes the color but also partially removes the following substances:

- Colloids
- Unpleasant taste
- Odor-causing substances
- Foam-causing substances (mainly, saponin)

The three most important factors affecting the decolorization efficiency are:

- The amount of contact time between the juice and carbon
- The amount of activated carbon used
- The temperature of the juice

In sugar factories, activated carbon is used in powder or granular form.

Powder-activated carbon (PAC) is usually used for the decolorization of thick juice or standard liquor because these in-process products have the right dry substance content (55 to 65%) for PAC operation. PAC application removes about 50% of the colorants. Their decolorization efficiency depends on their characteristics:

- 90% of carbon particles should pass a 325 U.S. screen (90% should be less than 0.075 mm)
- Surface area
- Pore size

The decolorization process is provided in a tank. The process consists of the following steps:

1. The juice is heated to about 80°C.
2. PAC is mixed as slurry with the feed in the mixing tank.
3. The process continues with a slow mixing for about 10 minutes.
4. The juice is filtered with a pressure-leaf filter (a filter aid is required for precoating).
5. The filtration is sweetened, and the filter residue is discarded or used to coat pressure filters.

The amount of PAC used varies from 0.05% to 0.2% on *DS* of the feed, depending on the feed color. To use PAC more economically, usually a lighter juice is treated first and then a darker one, after which the filter residue is discarded.

In comparison with the decolorization with granular activated carbon, the installation of a PAC decolorization process has the advantage of requiring low capital without major plant changes. The decolorization with granular carbon requires a larger capital investment, but the operating cost is lower because granular carbon can be regenerated and reused. (Note: Regeneration of PAC in kilns is not possible because the carbon will burn.)

Granular-activated carbon (GAC) is used in fixed-bed or continuous-bed columns to decolorize thick juice or standard liquor. In a fixed-bed system, a set of three columns is usually used. Two columns are usually in service and the third one is in regeneration mode. The columns are about 10 to 12 m (30 to 40 ft) in height and about 3 m (10 ft) in diameter. Each column contains about 100 m^3 (3500 ft^3) of carbon.

The decolorization station consists of two or more columns. The syrup flows upward against carbon movement (countercurrent operation). The carbon is partly discharged at the bottom of the column to be regenerated. Two columns about 15 m (40 ft) high are used for decolorizing the product. In a typical two-column system, the process consists of the following steps:

1. The feed is diluted on line to about 60% *DS* if it is concentrated.
2. The juice is heated to about 85°C.
3. The heated juice is pumped, at about 0.3 m^3/min (11 ft^3/min), to the bottom of each column.
4. The juice moves upward through the carbon, and leaves the column at the top. Therefore, the lower part of the beds becomes exhausted first.

5. The lower part of the carbon (about 5 m^3, or 180 ft^3) is discharged from the bottom of one of the columns (at intervals of about four hours) and flows by gravity to a tank to be sweetened.
6. At the same time, an equal amount of reactivated carbon, mixed in the juice, is pumped into the top of the column from which the exhausted carbon was discharged.
7. The exhausted carbon is pumped to the sweetening tank, where the carbon is drained by rinsing and becomes ready for reuse.
8. The sweeten-off carbon slurry is discharged into a dewatering tank, where the water is pushed out by air.
9. The drained carbon, which has about 40% moisture, is fed by a conveyor to the reactivation kiln, where the color is burned.

Reactivation (regeneration) of GAC usually takes place in a gas-heated kiln at 900 to 1000°C. The multiple-hearth kiln consists of a vertical cylindrical shell, 1 to 4 m (3 to 13 ft) in diameter, lined with refractory bricks. There are 6 to 10 circular hearths stacked one above the other inside the kiln. In most cases, a small quantity of excess air and steam is passed through the kiln.

GAC operation has an advantage over PAC because the exhausted GAC can be reactivated (regenerated) in kiln about 25 times. It also has higher decolorizing efficiency and lower operating cost compared with PAC operation.

NOTES

- **Some factories have a contract with a carbon supplier to transfer the exhausted carbon to the supplier site for reactivation. The advantage is these factories do not need a kiln for regeneration.**
- **Some sugar factories use bone char for decolorization. Bone char is animal bone in granular form and is produced from cattle bones heated to 700°C. Calcium phosphate is the main substance of the bone char (about 90%), and the rest is carbon (10%). Due to its high surface area and phosphate content, bone char can absorb color as well as organic and inorganic components, making it a good color remover and a purity enhancer. Its dosage is about 20% on the solids of the juice. The purest syrup is fed to the column filled with regenerated bone char, and as the decolorization performance declines, less pure solutions follow. The bone char usually gets exhausted in four to five days of operations depending on the color contents of the syrup. For regeneration of the exhausted bone char, it is washed with hot water to remove the calcium sulfate and then regenerated in a kiln at 500 to 550°C. Bone char has low absorbent capacity, and therefore the volume of bone char and the columns required are large. Bone char operation is costly, space-consuming, and labor-intensive.**

JUICE SULFITATION

Juice sulfitation is the process of adding **sulfur dioxide** (SO_2) to the juice to reduce color and prevent color formation in the next steps of operation. SO_2 inhibits the **browning** (Maillard) reaction that forms coloring compounds during evaporation and crystallization. It is usually used before the evaporation (to thin juice) and crystallization (to thick juice or standard liquor) process at about 30 ppm on juice. It is used also as a biocide to kill microorganisms in the diffuser (see Station 7).

When SO_2 is added to the juice, **sulfurous acid** (H_2SO_3) is formed:

$$SO_2 + H_2O \rightarrow H_2SO_3$$

Sulfurous acid is a strong reducing agent, which creates the following advantages:

- **Reduces color:** Sulfite ions act as a bleaching agent to reduce the color. Color is reduced also by attaching hydrogen ions of sulfurous acid to the colorant structure. Because of both reactions, a color reduction of up to 30% is achieved.
- **Prevents color formation:** Sulfite ions are effective in blocking color-forming **Maillard reactions**. This protects the juice from darkening during the next processing steps, when the juice is processed at high temperatures. The sugar produced from a SO_2-added juice forms less color during storage.

Although sulfitation has two major advantages, it also has two minor disadvantages:

- **Lowers alkalinity:** Sulfitation lowers the juice alkalinity (by about 0.01%) by replacing alkaline carbonate with neutral sulfate:

$$K_2CO_3 + H_2SO_4 \rightarrow K_2SO_3 + CO_2 + H_2O$$

- **Reduces pH:** Sulfitation usually reduces the pH value (by about 0.5 units at normal usage).

(Note: The colorants belong to nonsugars, but sulfitation does not raise the purity of the juice because the decolorized substances remain in the solution.)

Sulfur dioxide is usually used in thin juice before evaporation and in standard liquor before crystallization. The amount of usage is about 50 ppm for thin juice and 25 ppm for standard liquor (see the following Notes). The addition of SO_2 to the thin juice before evaporation reduces the color increase between the thin juice and the thick juice during evaporation. A 30% color increase during evaporation is expected when the juice is not treated with SO_2. With SO_2 addition, the color increase reduces to about 20%.

NOTES

- **FDA (Food and Drug Administration) requires that the amount of sulfur dioxide in food products, including granulated-refined sugar, not exceed 20 ppm. Other standards, such as the Codex Alimentarius Standard, permit a maximum level of 15 ppm. Therefore, sugar should be tested routinely for sulfur dioxide content in the laboratory to adjust the amount of sulfur dioxide used during operation.**
- **Sulfitation should always take place before filtration to remove the calcium sulfate precipitate from the juice during filtration.**
- **Sulfitation should always take place after thin-juice softening because of the risk of calcium sulfite precipitation and resin pore blockage during softening. In factories equipped with thin-juice softening (Chapter 6), it is recommended that the juice not be sulfited before the softening operation. The sulfur can be added to thick juice and standard liquor to decrease their color.**

Production of Sulfur Dioxide

Some sugar factories produce their required sulfur dioxide by burning sulfur in a rotary **sulfur stove**. However, most factories buy **liquid sulfur dioxide** because sulfur burning is difficult and can be a health hazard if all safety precautions are not followed.

Sulfur burns in the stove according to the following equation:

$$S + O_2 \rightarrow SO_2$$

When excess air is used, the equation proceeds further to produce SO_3:

$$2SO_2 + O_2 \rightarrow 2SO_3$$

The SO_3 is not desired because it produces sulfuric acid in alkaline solution. SO_2 produces sulfurous acid (H_2SO_3), which is used as a bleaching agent for juice decolorization. Newly made nozzle burners with air controls can adjust to the amount of air usage and minimize the formation of SO_3. Sulfur dioxide produced in the stove is usually introduced to the juice with a steam injector or a gas pump.

A problem that may occur is the sublimation of sulfur into the gas in the gas line. You can minimize sublimation by keeping the lines cool. **Sublimation** is the direct change from a solid state to a gas state without passing through the intermediate liquid state.

As stated, most sugar factories now purchase liquid SO_2, which comes in a tank car and is loaded into the steel sulfur-dioxide tank. The sulfur-dioxide tank is installed outside the main building, and the SO_2 is pumped to the required stations. Usually, one line is introduced to the diffuser, one to the thin juice line going to the evaporator station, and one to the standard liquor line going to the white pans. By having separate lines on thin juice and standard liquor, you have the opportunity to use the gas in only one or in both.

Sulfur Dioxide Properties and Precautions

Sulfur dioxide is colorless and smells like a burnt match when it burns. Sulfur dioxide is **poisonous** and can kill people outright at 500 ppm. Sulfur dioxide is easily dissolved in water and the dissolving process is an **exothermic reaction** (releasing-energy reaction). Sulfur dioxide can be liquefied under moderate pressure at room temperature. Liquid sulfur freezes at –73°C (–99.4°F) and boils at –10°C (+14°F) at atmospheric pressure. It is important to keep the sulfur dioxide above its freezing point.

NOTE

- **Care must be taken when handling sulfur dioxide. If you start coughing because of sulfur inhalation, leave the area and breath some fresh air.**

OPERATING PROBLEMS

Some of the **problems** you can encounter in the juice decolorization process are listed here.

Low Decolorization Efficiency

The usual causes of low efficiency in decolorization by resin or carbon are as follows:

- **High dry substance:** Feed with a high dry substance content (more than 65%) decreases decolorization efficiency.
- **Low temperature:** Low temperature (lower than 80°C) feed reduces efficiency.
- **Low pH:** Feed with a low pH (lower than 8.3) reduces decolorization efficiency.
- **Low-purity feed:** Feed with low purity (lower than 85%) decreases decolorization efficiency. In case of processing low-purity feed, decolorizing adsorbents (resin or carbon) adsorb not only color but also the nonsugars, lowering the efficiency of the decolorization.
- **High invert sugar:** Feed with a high invert-sugar content decreases decolorization efficiency because a direct relationship exists between color formation in the juice and its invert-sugar content.
- **Unclear feed:** Unclear juice (juice not filtered properly) reduces decolorization efficiency because unclear juice may contain colloidal substances that plug the adsorbent pores, causing low decolorization efficiency.
- **Underlimed juice:** Underlimed juice (juice produced from insufficient retention time or low temperature during purification) is subject to more color change during evaporation, causing low efficiency during decolorization.

CHAPTER 3

SECTION 15

JUICE STORAGE

SUBSECTION

- Storage Conditions
- Process Accounting of Juice Storage
- Economy of Juice Storage
- Equipment Requirement
- Crystallization of the Stored Product

ABOUT THIS SECTION **This section** familiarizes you with **Station 15** (Juice Storage) of sugarbeet processing. Station 15 (see Figure 3.15.1) includes the tanks and other equipment for storing juice. Some beet-sugar factories are equipped with this station and can process sugarbeet to more than their normal capacity. They send the excess juice (the juice produced beyond the sugar-end capacity) to juice storage to be processed later during the beet intercampaign. Juice storage is mostly used in factories with a higher beet-end capacity than the sugar-end can handle or in factories that plan to expand their beet-end capacity. The stored product is processed in the sugar-end into granulated-refined (GR) sugar during the **juice campaign** (the period between two beet campaigns during which the stored product is processed). The products usually stored are thick juice, standard liquor, and extract (product of molasses desugaring by chromatographic [MDC] process).

In this section, you learn the advantages of storing juice, its economy, and how to use **granulated equivalent** (GE) to compare the storing costs in two systems: juice storage in a tank and sugar storage in a silo. In other words, you will find the answer to an important question: "Which system offers lower cost per ton of sugar stored?"

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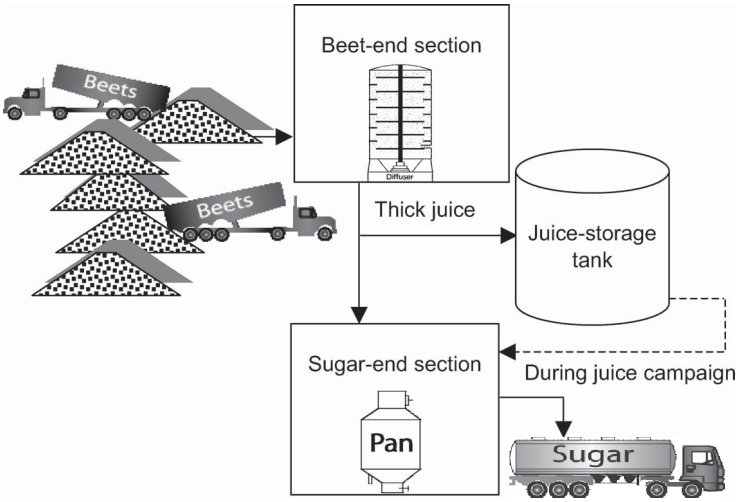


Figure 3.15.1 Flow diagram of juice storage (Station 15)

Juice storing (juice storage) is the process of storing juice during the beet campaign in the juice storage to increase sugar-end capacity of the factory. Thick juice and standard liquor are the in-process products usually stored in storage. If the factory is equipped with molasses desugaring by chromatographic process (**MDC process**), **extract** (the product of the MDC process) is stored as well. The stored products are processed later during the juice campaign. The main purposes of juice storage are the following:

- Increase beet-end capacity for less investment
- Increase the equipment-utilization period
- Increase the sugar production period
- Decrease the beet-campaign period
- Decrease sugar loss in sugarbeets
- Save on the sugar silo's capacity

Today, juice storing in the tank is part of normal operations of many beet factories because of its economical advantage over sugar storing in the sugar silo, as discussed later under economy of juice storing. Juice storage puts the factory into a two-mode operation: beet and juice campaign, with a capability of processing the juice anytime during the beet intercampaign (see the Note).

NOTE

The flexibility of moving the juice campaign to a desired time (usually right after finishing the beet campaign or before starting the next campaign) is helpful because it can be managed by sugar silo requirements. However, the juice stored is usually processed quickly, resulting in a short juice campaign (two to four weeks depending on the availability of the juice).

STORAGE CONDITIONS

Juice storage requires a high level of care before and during storage. The following factors have to be considered:

- **Dry substance** (*DS*)**:** The *DS* of the stored product should be between 67 and 72%.
- **pH:** The pH of the product should be between 8 and 9.5 to reduce bacterial activities and sugar loss (in the case of low pH, adding 0.5 kg NaOH per ton of juice can increase the pH of the product by about one unit).
- **Purity:** The purity of the product should not exceed 92% because products with high-sugar content have more tendency to form crystals during storage.
- **Temperature:** The product should be cooled to around 30°C before being stored.
- **Filtration:** The product should be filtered to remove microbes not killed by heat and to filter out small sucrose particles to avoid **nucleation** (the formation of crystals with or without seeding) in the tanks or pipes.
- **Cleanliness:** Tanks for storage should be cleaned and well sterilized with a suitable biocide (chemicals for killing microbes) before use to prevent inversion of sucrose during storage.

In a conventional factory (a factory without the MDC process), the in-process product usually stored is thick juice or standard liquor. As candidates for storage, thick juice and standard liquor, each has one advantage over the other:

- Thick juice usually has a lower purity than standard liquor, which is an advantage because it decreases the chance of self-crystallization during storage.
- Standard liquor can be produced at the required concentration for safe storage without additional evaporation.

NOTE

If the factory is equipped with thin-juice softening (see Chapter 6) and molasses desugaring by chromatographic (MDC) process, extract storage can be better justified, compared to thick juice and standard liquor. This is because an extract contains very low hardness (limesalts), which puts the precipitation on tanks and pumps in the minimal range. The extract usually contains a purity of 90 to 92% and is concentrated to approximately 70% *DS* before being stored.

PROCESS ACCOUNTING OF JUICE STORAGE

The section helps clarify the process accounting of juice storing. To better understand the subject, let us study the process in the context of the following example.

EXAMPLE 1

Given:
A factory, with a capacity of 6000 t beets per day, plans to install juice-storage equipment to increase its beet-end capacity by 500 t/day. The other given data are as follows:

Diffusion juice *DS*	15.0%
Thick juice *DS*	67.5%

Thick juice purity	89.5%
Diffusion draft (D)	120%
White (A) massecuite purity	92.5%
High-green (A) syrup purity	84.8%
Molasses purity (P_M)	60.0%
Beet-campaign period	133 days

Calculate:

Amount of thick juice produced from 500 t beets

Storage tank requirement

Sugar-end recovery

Juice-campaign period (days)

Granulated-refined sugar produced

Ton sugar/ton juice (granulated equivalent)

The amount of thick juice (m_{TJ}), produced from 500 t beets (m_B = 500 t), can be calculated by using the dry substances of thin juice (DS_1) and of thick juice (DS_2), and the diffusion draft (D):

$$m_{TJ} = m_B \times \frac{D}{100} \times \frac{DS_1}{DS_2} = 500 \times \frac{120}{100} \times \frac{15.5}{67.5} = 137.8\,\text{t/day}$$

At a thick juice DS of 67.5, the density (d) of thick juice is 1.32 t/m^3 (from the density table in the Appendix). The thick-juice volume is

$$v = \frac{m}{d} = \frac{137.8}{1.32} = 104.4\,\text{m}^3\text{/day}\ (3686\,\text{ft}^3\text{/day})$$

$$104.4 \times 135 = 14094\,\text{m}^3\text{/135 days}\ (497659\,\text{ft}^3\text{/135 days})$$

The tank volume required for storing 1 t of juice is

$$\frac{104.4}{137.8} = 0.75\,\text{m}^3\text{/t juice}$$

14094 m^3 of juice needs an average tank 40 m (120 ft) in diameter and a height (h) of

$$h = \frac{v}{\pi(d/2)^2} = \frac{14094}{3.14(40/2)^2} = \frac{14094}{1256} = 11\,\text{m}\,(33\,\text{ft})$$

The sugar-end recovery (R) from the stored thick-juice operation can be calculated by using the purity of thick juice (P_J) and that of molasses (P_M) as follows:

$$R = \frac{(P_J - P_M)}{P_J(100 - P_M)} \times 10000 = \frac{(89.5 - 60.0)}{89.5(100 - 60)} \times 10000 = 82.4\%$$

Knowing that the sugar-end can handle juice from 6000 t beet a day, the juice campaign period (in days) can be calculated based on the slice ratio, because of the similarity of sugar-end operations during the beet and juice campaigns:

$$137.8 \times \frac{500}{6000} = 12 \text{ days}$$

With the purity of white massecuite (P_2) and that of high-green syrup (P_1) known, the crystallization efficiency (nonsugar elimination, *NSE*) (see the Appendix) is

$$NSE = \frac{(P_2 - P_1)}{P_2(100 - P_1)} \times 10000 = \frac{(92.5 - 84.8)}{92.5(100 - 84.8)} \times 10000 = 54.8\%$$

The juice produced during the beet campaign is

$$137.8 \times 135 = 18603 \text{ t}$$

The granulated-refined sugar produced during the juice campaign is

$$18603 \times \frac{54.8}{100} = 10194\,t$$

Finally, the amount of sugar produced from 1 t of juice, called the **granulated equivalent** (*GE*) is

$$GE = \frac{10194}{18603} = 0.55t$$

ECONOMY OF JUICE STORAGE

From an economical point of view, it is important to compare the storage cost in the two systems: juice storage in a tank and sugar storage in a sugar silo. The comparison follows:

- **Operating costs:** The expensive part of sugar storage depends on how the sugar is handled and stored in a sugar factory. The variable costs for processing juice can be from $15 to $40 per ton sugar depending on the facility (Carlson 2005). The variable costs for sugar storage are much lower.
- **Capital investment:** The capital investment in juice storage is considerably lower than the costs of expanding the sugar-end capacity because the equipment at the sugar-end (pan crystallizer, centrifuges, etc.) is more costly than the equipment required for juice storage. In fact, juice storage expands the utilization period of the sugar-end equipment. Regarding the capital requirement for the two storage systems, a steel Weibull-type silo requires as much as $5 million for storage of 30000 t of sugar (about $170/t sugar). Juice-storage tanks and auxiliaries for the storage of granulated equivalent (about 60000 t of juice) cost about $1.5 millions (about $50/t sugar), which is lower than the sugar silo by 70%.
- **Labor costs:** Labor costs of the sugar-end during the juice campaign are higher than during the beet campaign because the required labor force is greater. Sugar storage in a silo requires about $1.5 labor per day per ton sugar (Van der Poel et al. 1998) and juice storage costs less.

The preceding comparison proves that the return on investment (ROI) of juice storage is considerably shorter than expanding the sugar-end and sugar silo to handle the juice produced from extra sugarbeet processed during the beet campaign. In other words, *juice storage in a tank offers lower cost per ton of sugar stored than sugar storage in a silo.*

EQUIPMENT REQUIREMENT

The **main equipment** required for juice storage is a storage tank (see Figure 3.15.2). Stored juices in sugar factories are usually heavy compared to other materials. For example, a juice with 70% *DS* has a density of 1330 kg/m^3 (83 lb/ft^3), which is heavier than fuel oil (which has a density of about 900 kg/m^3). Therefore, the tanks used for juice storing must be able to withstand the stress placed on them. Welded **fixed-roof tanks** 40 to 48 m (132 to 155 ft) in diameter and 15 to 18 m (50 to 60 ft) in height with a holding capacity of 25000 to 43000 t are usually used for juice storage. The bottom and the top of the tank are cone shaped. The height of the bottom cone is about 1 m (3 ft). The roof is supported by several legs. The input product is pumped to the bottom of the tank, and the output juice is pumped from a well about 1 m (3 ft) in diameter by 1 m deep located at the low point of the tank.

The disadvantage with a fixed-roof tank is that a large juice surface is exposed to the atmosphere, causing dilution of the stored product at the surface because of condensation. To prevent dilution, one of the following is recommended:

- Install fans at the top of the tank to move the air space
- Use food-grade oil to cover the surface of the stored product
- Replace the roof of the tank with a floating roof

Floating-roof tanks provide the best storage conditions. Under normal conditions, juice can be stored in a floating-roof tank for more than three years without a loss in quality. The most important factor in designing a floating roof is maintaining clearance between the floating roof and the tank's wall to ensure the following:

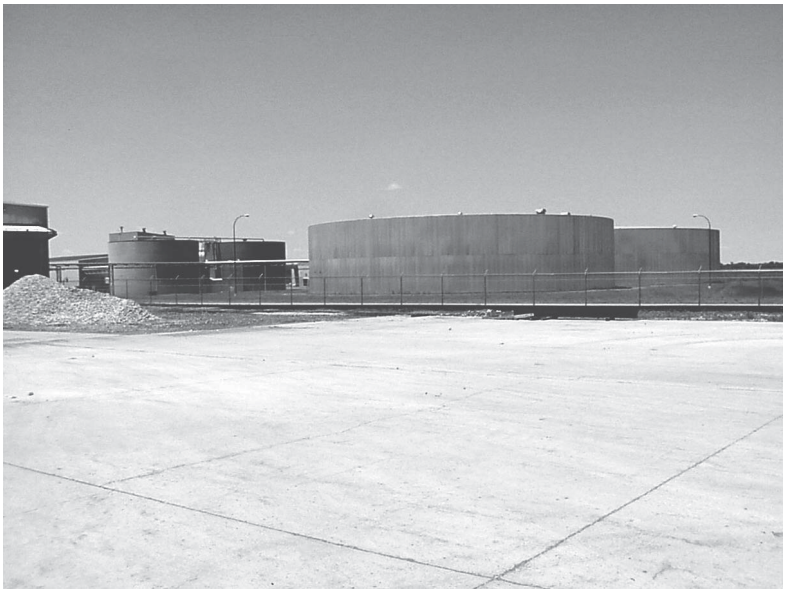


Figure 3.15.2 Juice-storage tanks

- Smooth movement of the roof up and down
- Sealing between the roof and side wall to prevent leakage

Coating the inside of the tank with special food-grade paint protects the juice from any attack from the surface of the tank.

Besides storage tanks, the juice-storage station contains the following auxiliary equipment:

- **Filters:** Pressure-leaf filters (see Station 11) are usually used before the product is stored.
- **Cooler:** Plate heat exchangers (see Station 13) are usually used.
- **Sampling points:** The tank should be equipped with a short pipe and a locking valve in every 1.5 to 2 m (4.5 to 6 ft) for sampling. This enables the operator to get a sample from different levels of the stored product.
- **Measuring device:** Used to measure the tank's content (see the following Note)
- **Equipment for adjusting pH**
- **Juice pumps**

NOTES

- **For maximum accuracy, the inventory of the stored juice should be based on weight, not volume, if possible. Flowmeters are not accurate enough for measuring a tank's content. If the factory does not have weighing scales and uses manometers to measure the volume, the mass of the stored juice is calculated per unit of height of the tank.**
- **Usually, the factory chemist keeps inventory of the tanks' contents. In the following example, the mass of the juice at increments of 2.5 cm (1 in.) is calculated. All calculations are given in both metric and English measurements.**

EXAMPLE 2

Given:

Tank diameter (d)	48 m (160 ft)
Tank height (h)	16 m (53 ft)
Cone height	1 m (3 ft)
Juice *DS*	70%
Juice density	1.33 t/m^3

Calculate:

Volume of each 2.5 cm (1 in.) above the cone

Weight of each 2.5 cm above the cone

Total volume of the juice in the cone

Total mass of the juice in the cone

Total tank capacity

We can calculate the volume (v) and mass (m) of each 2.5 cm of juice above the cylindrical cone as follows:

$$v = \pi\left(\frac{d}{2}\right)^2 \times h = 3.14\left(\frac{48}{2}\right)^2 \times \frac{2.5}{100}\ 45.2\ \text{m}^3/2.5\ \text{cm}\ (1596\ \text{ft}^3/\text{in.})$$

$$m = v \times d = 45.2 \times 1.33 = 60.1\ \text{t}/2.5\ \text{cm}\ (67.3\ \text{t/in.})$$

The total volume of the juice in the cone is

$$v = \frac{\pi(d/2)^2 \times h}{2} = \frac{3.14(48/2)^2 \times 1}{2} = \frac{1808.6}{2} = 904.3\ \text{m}^3 (31922\ \text{ft}^3)$$

The total mass (m) of the juice in the cone is

$$m = 904.3 \times 1.33 = 1203\ \text{t}\ (1347\ \text{short tons})$$

The total mass of the juice in the cylindrical part is

$$m = 60.1 \times 16 \times \frac{100}{2.5} = 38464\ \text{t}\ (43080\ \text{short tons})$$

The total tank capacity is

$$m = 1203 + 38464 = 39667\ \text{t}\ (44417\ \text{short tons})$$

CRYSTALLIZATION OF THE STORED PRODUCT

During the juice campaign, the stored product is processed in the sugar-end into granulated-refined (GR) sugar and molasses. The sugar-end operation during this period is similar to the beet campaign with one major difference: there is no thin-juice evaporation during the juice operation. To supply heat to the sugar-end, one of the following is practiced:

- Condensate (some condensate is always stored before the beet campaign ends) is evaporated in the evaporators to produce vapor for the pan crystallizers.
- The stored product is diluted to 30 to 40% *DS*, filtered, evaporated to reach a concentration of about 75% *DS*, and sent to the sugar-end. The vapor produced in the evaporators is used in the sugar-end.

Although the second option looks like you are doing the job twice, it is the better choice as far as heat economy and technology are concerned (Van der Poel et al. 1998).

CHAPTER
3

SECTION 16
SYRUP CRYSTALLIZATION

SUBSECTIONS

- Sucrose Solubility
- Supersaturation and Viscosity, Opposing Forces
- Crystal Content
- Crystallization in a Beet-Sugar Factory
- Batch Crystallization
- Crystallization Control and Automation
- Sugar Boiler
- Seed Preparation
- Crystallization Theory
- Crystallography of Sucrose
- Crystallizers
- Operating Problems

ABOUT THIS SECTION **This section** details **Station 16** (Syrup Crystallization) of sugarbeet processing. Station 16 (see Figure 3.16.1) includes the crystallization equipment, the most delicate of the production process, to produce sugar. To recover the maximum amount of sugar from thick juice, beet-sugar factories usually use a **three-stage crystallization** process. In the first stage (called the white side), market-quality sugar is produced. In the second and third stages (called the raw side), raw sugars are produced, which are dissolved in the thick juice to produce **standard liquor** (the feed for the first stage of crystallization). The mother liquor of the third stage is called **molasses**, which is difficult to crystallize because of its high viscosity and high nonsugar content. Therefore, molasses is separated from the system to prevent nonsugar (impurity) accumulation.

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Because the crystallization process is an extensive subject, Station 16 explains only the first stage (white side) of it, in which market-quality sugar is produced. Subjects related to raw side (high-raw and low-raw crystallization) and molasses exhaustion (production of molasses with satisfactory purity) are discussed in the next section (Station 17).

Rock-candy making is a simple example of the crystallization process, in which sugar (sucrose) crystals are formed and grown in a supersaturated solution. When more sugar crystals are added at a high temperature to a saturated sugar solution, it becomes **supersaturated** (overdissolved). The overdissolved sugar is crystallized when the solution becomes cold. The product is called rock candy, which you can make at home. Here is how:

- Heat water in a small cooking pot until the water is close to boiling.
- Pour sugar while stirring the solution and continue until no more sugar dissolves. The solution is now **saturated**.
- Increase the temperature slowly until the liquid boils and add more sugar to dissolve.
- Add more sugar (one spoonful at a time) until it can be dissolved, indicating clear solution with no undissolved sugar crystals. By now, the solution is **supersaturated**.
- Remove the pot from the heat.
- Tie one side of a string to a weight and the other side to a long pencil. Put the weight almost to the bottom but not touching.
- Support the string with the long pencil across the rim of the pot and leave the pot undisturbed.
- As it cools, the overdissolved sugar starts to crystallize on the string until the liquor becomes saturated. Because the pot is uncovered, the water evaporates, and the liquor becomes supersaturated again, causing additional molecules of sugar to attach themselves to the existing crystals. The change from saturation to supersaturation, and vice versa, continues until the crystals grow to the desired size.
- In about a week, in the pot there will be large rock candy surrounded by a liquor, known as **mother liquor** that can be washed from the crystals by rinsing with a little cold water.

Crystallization (**boiling**, in the sugar boiler's language), in sugar technology, is a mass transfer of sugar molecules from the syrup to the solid particles (seeds) to form crystals. Crystallization leaves impurities in the syrup (known as **mother liquor**). Crystallization is one of the most effective separation techniques, providing an impurity-elimination effect of up to 99.9%. In other words, impurities are excluded from the crystallization process and pure solute is the only substance that crystallizes (granulated-refined sugar contains above 99.9% sucrose and raw sugar 96 to 99%). Crystallization occurs when the syrup is supersaturated and other conditions are met (more discussion about it later in this section).

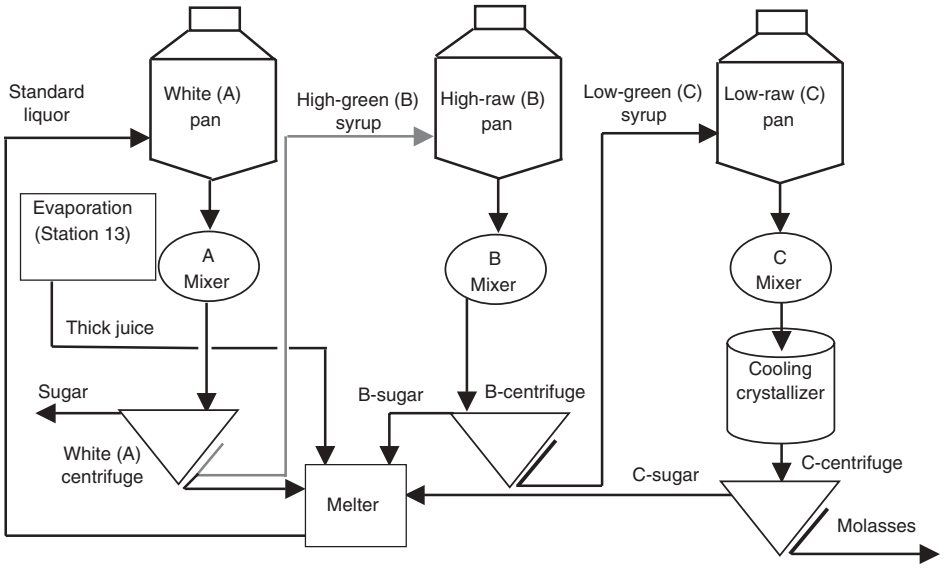


Figure 3.16.1 Three-stage crystallization (Station 16)

From a sugar-technology point of view, crystallization is well performed when uniform crystals of high purity are produced. Crystallization occurs when solute (in our case, sucrose) moves from a supersaturated solution (in our case, impure sucrose solution) and attaches to the crystal structure. This definition includes three classes of crystallization:

- Flashing crystallization (crystallization by evaporation under vacuum)
- Evaporating crystallization (crystallization by evaporation)
- Cooling crystallization (crystallization by cooling)

Sugar factories use flashing and cooling crystallization. In flashing crystallization, water is evaporated under vacuum (at about 74°C) and seed is applied, causing crystallization to occur because the water present in the mother liquor is not enough to hold sugar molecules. In cooling crystallization, the temperature of the **massecuite** (explained later) is lowered to about 40°C, causing crystallization to occur because the water present in the mother liquor holds less sugar (*solubility decreases with decreasing temperature*).

In flashing crystallization, the presence of a vacuum decreases the boiling point (*BP*) of the syrup, preventing the inversion of sucrose and saving energy. The general concept of crystallization is discussed here; cooling crystallization is explained in the following section (Station 17).

The main purposes of crystallization in the sugar factory are to produce:

- Sugar with the highest possible quality, at high efficiency and low cost
- Molasses with the lowest possible purity (called exhausted or satisfactory molasses)

In sugar terms, the product of the crystallization process is called **massecuite** (known as **mass** to sugar technologists), which is a semisolid mixture consisting of crystals and the surrounding

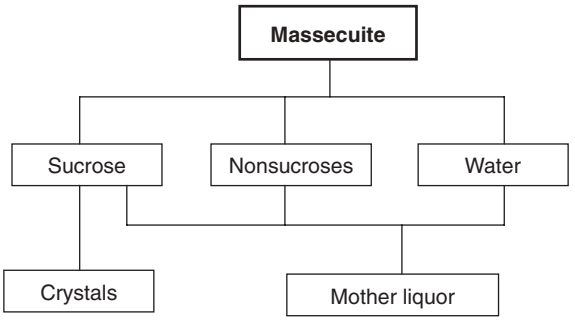


Figure 3.16.2 Massecuite composition

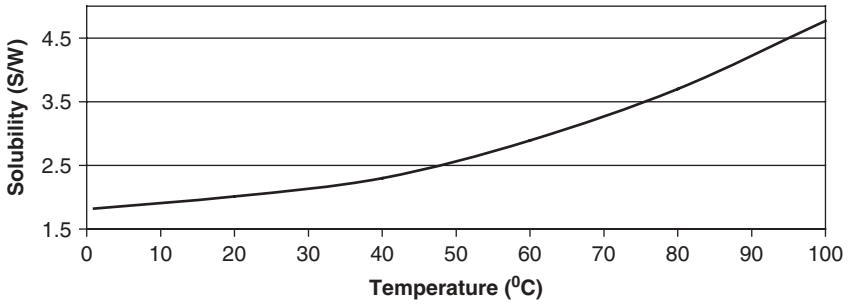


Figure 3.16.3 Sucrose solubility in water at different temperatures

solution around the crystals, called **mother liquor** (see Figure 3.16.2). In other words, mother liquor is the liquor from which crystals are formed that stays with the crystals until they are separated by centrifuging. Therefore, the term mother liquor is used later in this section even when no crystals are in the liquor yet (before seeding).

SUCROSE SOLUBILITY

Sucrose solubility in pure water is the maximum amount of sucrose (sugar) that can be dissolved in 1 g of water (g sucrose/g water) at a given temperature. Sucrose is highly soluble in water. At 100°C, almost five parts sucrose can be dissolved in one part water to produce a saturated solution. Sucrose solubility in pure water depends only on the temperature of solution and increases considerably with rising temperature (see Figure 3.16.3).

Sucrose solubility in impure (technical) **solutions** is defined as the maximum amount of sucrose (in grams) that can be dissolved in 1 g of solution at a given temperature. Although sucrose solubility in water is dependent only on the temperature, its solubility in impure solutions depends on three factors:

- Temperature
- Nonsucroses content
- Nonsucroses characteristics

In sugar technology, **crystallization** is based on the solubility of sucrose in impure solutions with different concentrations and temperatures. At the same temperature, the concentration of sucrose in solution determines the type of solution, which may be *unsaturated*, *saturated*, or *supersaturated*. Solutions are differentiated from each other based on solubility.

In an **unsaturated** (undersaturated) **solution**, the solubility of solute (in our case, sucrose) has not reached its maximum limit yet. For example, a sucrose solution containing 150 g sucrose in 100 g water at room temperature (20°C) is an unsaturated solution.

In a **saturated solution**, the solubility of solute has reached its maximum limit. If more sucrose is added to an undersaturated solution and stirred to dissolve it, eventually the solubility limit is reached, and more sucrose cannot be dissolved because no free water molecules are left in the solution to interact with sucrose molecules. In a saturated sucrose solution at a given temperature, sucrose will neither crystallize nor dissolve (*saturated solution is in equilibrium condition*).

In a **supersaturated** (oversaturated) **solution**, the solubility of solute has passed its maximum limit. In other words, a supersaturated solution contains more solute than normally can be dissolved in a saturated solution. In a supersaturated sucrose solution, sucrose will crystallize by a disruption such as seeding (*a supersaturated solution is not in an equilibrium condition*).

Thus far, you have learned that the main difference between undersaturated, saturated, and supersaturated solutions is their sugar concentration (an undersaturated solution contains the least amount of sugar, and a supersaturated solution contains the highest). Next, we compare sucrose solubility in these solutions and find out the effects of impurities (nonsucroses) on sucrose solubility. In other words, sucrose solubility in pure water is compared with sucrose solubility in impure solutions. The comparisons can be expressed by three **coefficients** (a *dimensionless* ratio that compares the change of a variable with respect to other variables):

- Solubility coefficient
- Saturation coefficient
- Supersaturation coefficient

Solubility Coefficient

Solubility coefficient (K_S) compares sucrose solubility in pure water (part sucrose to each part of water) at different temperatures. In other words, K_S (S stands for solubility) indicates the **sucrose-to-water ratio** (S/W) at a given temperature. For example, the maximum amount of sucrose that can be dissolved in 1 g water at 20°C is 2.0 g (2 parts sucrose to 1 part of water, or $k_S = 2.0$). This is a 66.7% (by mass) sucrose solution (see Figure 3.16.3). K_S is given as

$$K_S = \frac{S}{W} = \frac{S}{100 - S} \tag{3.16.1}$$

EXAMPLE 1

Given:

A pure sucrose solution has the following properties:

Temperature	40°C
Sucrose content (S)	70.1% (by mass)

Calculate:

Solubility coefficient of sucrose (K_S)

Whether or not the solution is saturated at this temperature

$$K_S = \frac{S}{100 - S} = \frac{70.1}{100 - 70.1} = 2.34$$

This means that there is 2.34 parts of sucrose for each part of water (the sucrose-to-water ratio is 2.34) in this solution. This solution is saturated because at 40°C, the K_S equals to 2.34 (from Table A.2 in the Appendix). In other words, the solution is saturated with sucrose in the ratio of 2.34 to 1, and from this point, no more sugar can be dissolved unless the temperature is increased. For example, if the temperature is increased to 50°C, the sucrose solubility (S/W ratio) will increase to 2.59 g/g.

Over the years, sugar scientists have tried to find a unique expression that correlates the effect of **temperature** (T) on **solubility** (S). Sucrose solubility data, given in Table A.2 in the Appendix, are calculated based on the Vavrinecz equation and the ICUMSA adaptation (both equations are given in Bubnik et al. 1995):

$$S = 64.447 + 0.08222T + 1.6169 \times 10^{-3} T^2 - 1.558 \times 10^{-6} T^3 - 4.63 \times 10^{-8} T^4 \% \qquad (3.16.2)$$

For temperatures between 100 and 125°C, sucrose solubility can be calculated based on the Smelik equation (the equation is given in Bubnik et al. 1995):

$$S = 71.0615 + 5.3625 \times 10^{-2} T + 6.55303 \times 10^{-4} T^2 \% \qquad (3.16.3)$$

EXAMPLE 2

Given:

Water at 20°C temperature

Calculate:

Sucrose solubility (%)

Sucrose solubility (g/g)

$$S = 64.447 + 0.08222 \times 20 + 1.6169 \times 10^{-3} \times 20^2 - 1.558 \times 10^{-6} \times 20^3 - 4.63 \times 10^{-8} \times 20^4$$

$$S = 66.74\%$$

$$K_S = \frac{S}{100 - S} = \frac{66.74}{100 - 66.74} = 2.00\,\text{g/g}$$

Saturation Coefficient

Saturation coefficient (K_{IP}) compares sucrose solubility in an *actual impure solution* with a *pure saturated solution*, both at the same temperature. In other words, K_{IP} (I stands for impure and P for pure) indicates the effect of nonsucroses on sucrose solubility in impure sucrose solutions. K_{IP} is given as

$$K_{IP} = \frac{(S/W)_{\text{Act}}}{(S/W)_P} = \frac{K_{\text{Act}}}{K_P} \tag{3.16.4}$$

$(S/W)_{\text{Act}}$ Sucrose-water ratio in actual solution (g/g)
$(S/W)_P$ Sucrose-water ratio in pure saturated solution (g/g)

Some nonsucroses present in beet juice increase the sucrose solubility, and some such as invert sugar decrease it (this is the reason that invert sugar has tendency to slow down the crystallization process). The effect of nonsucroses on sucrose solubility depends on the amount of these nonsucroses (the purity of the solution) and their characteristics. Because of the difference in nonsucrose characteristics, syrups with different K_{IP} can hold different amounts of sucrose, affecting the rate of crystallization.

Figure 3.16.4 shows the typical sucrose solubility in beet juice for different purities at 75°C (the usual pan boiling temperature, under vacuum). (Sucrose solubility in an impure solution is greater than in pure solution when the purity of the solution is less than 93% and is smaller or the same when the purity is higher than 93%.) For example, the sucrose solubility at 75°C in pure water is 3.46 g per g water. In 80-purity syrup at the same temperature, its solubility is 3.48 (greater), and the solubility is 3.36 (the same) in 95-purity syrup.

NOTE

Most in-process products (juices, syrups, and liquors) of sugar processing have a purity below 93%. Therefore, increasing nonsucroses (decreasing purity) in these products increases sucrose solubility.

In the case of impure solutions, sucrose solubility can be calculated from the equation derived by Vavrinecz (the equation is given in Bubnik et al. 1995). (Note: The calculations become complicated when purity is below 70%.)

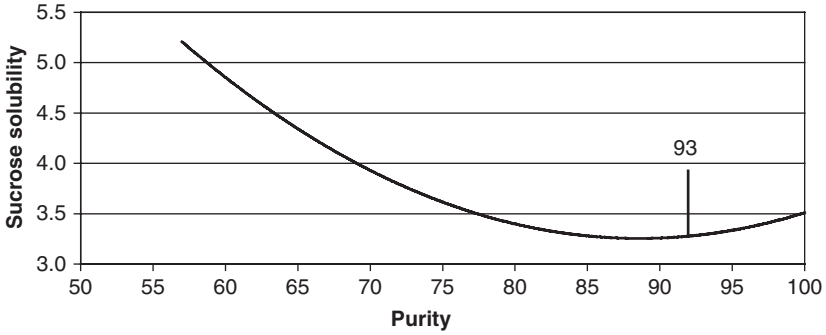


Figure 3.16.4 Effect of juice purity on sucrose solubility at 75°C

$$S = \left[0.178 \times \frac{NS}{W} + 0.820 + 0.180 \times \exp\left(-2.1 \times \frac{NS}{W}\right)\right] \times K_P\% \tag{3.16.5}$$

$$\frac{NS}{W} = \frac{DS - S}{100 - DS} \tag{3.16.6}$$

EXAMPLE 3

Given:

Standard liquor at the seeding point in the white pan has the following qualities:

Temperature	75°C
Dry substance (*DS*)	80.0%
Purity (*P*)	93.0%

Calculate:

Saturation coefficient (K_{IP})

$$S = \frac{DS \times P}{100} = \frac{80.0 \times 93.0}{100} = 74.4\%$$

$$\left(\frac{S}{W}\right)_{\text{Act}} = \frac{S}{100 - DS} = \frac{74.4}{100 - 80.0} = 3.72$$

According to Table A.2, sucrose solubility at saturation (K_P) and 75°C is 3.46, so K_{IP} is

$$K_{IP} = \frac{(S/W)_{\text{Act}}}{(S/W)_P} = \frac{3.72}{3.46} = 1.08$$

So the presence of nonsugars has increased the sucrose solubility by a ratio of 1.08 to 1.

Supersaturation Coefficient

Supersaturation coefficient (K_{SS}) compares sucrose solubility in an *actual impure solution* with an *impure saturated solution*, both at the same temperature (see the following Note). In other words, K_{SS} (*SS* stands for supersaturation) indicates if a solution is unsaturated ($K_{SS} < 1$), saturated ($K_{SS} = 1$), or supersaturated ($K_{SS} > 1$). K_{SS} is given as

$$K_{SS} = \frac{(S/W)_{\text{Act}}}{(S/W)_I} \tag{3.16.7}$$

$(S/W)_{\text{Act}}$ Sucrose-water ratio in actual solution (g/g)
$(S/W)_I$ Sucrose-water ratio in impure saturated solution (g/g)

NOTES

- **In the absence of automated control system, the sugar boiler estimates the supersaturation situation (refers to K_{SS}) by a proof test, also known as string proof (putting a small amount of syrup between two fingers and then moving the fingers apart until the string breaks) and by observation of the splash on the sight glasses at the syrup level. For example, the bubbles rise slowly and the splashes run down slowly as the syrup becomes more concentrated (K_{SS} increase to about 1.2) and ready for seeding. Later, in the Crystallization Control and Automation section, there will be more talk about how to automate the crystallization pans.**
- **The nominators of Eq. (3.16.4) and Eq. (3.16.7) are the same, but the denominators are not. In other words, numerically the difference between K_{IP} and K_{SS} is in their denominators. For calculation of K_{IP}, the denominator of Eq. (3.16.4) is taken from Table A.2 and for calculation of K_{SS}, the denominator of Eq. (3.16.7) is taken from Table A.3. Both tables are given in the Appendix). If you compare the result of Example 3 with Example 4, you can see the difference (in value) between K_{IP} and K_{SS}.**
- **The four main variables that affect K_{SS} are the dry-substance content (concentration), purity, temperature, and the saturation coefficient (K_{IP}) of mother liquor. Later, we will learn more about these parameters.**
- **The word oversaturation is used in sugar technology also to indicate the state of excess supersaturation ($K_{SS} > 1.5$), which means passing the safe (metastable) zone and entering the unstable (unsafe or labile) zone.**

EXAMPLE 4

Given:

Standard liquor at the seeding point in the white pan has the following qualities:

Temperature	75°C
Dry substance (*DS*)	80.0%
Purity (*P*)	93.0%

Calculate:

Whether the K_{SS} is right for seeding or not

This example is similar to Example 3 in which K_{Act} was calculated ($K_{\text{Act}} = 3.72$). According to Table A.3, sucrose solubility at saturation and at 75°C in syrup with a purity of 93 is 3.35, so K_{SS} is

$$K_{SS} = \frac{(S/W)_{\text{Act}}}{(S/W)_I} = \frac{3.72}{3.35} = 1.11$$

The time is right for seeding because the best K_{SS} recommended for seeding the white pan is between 1.1 and 1.2.

EXAMPLE 5

Given:

High-raw (B) massecuite is centrifuged, producing high-green (A-green) syrup with

Temperature	60°C
Dry substance (*DS*)	82.0%
Purity	72.0%

Calculate:

Supersaturation coefficient (K_{SS}) of the syrup

Sucrose content (S) of the syrup can be calculated from the purity formula:

$$S = \frac{DS \times P}{100} = \frac{82.0 \times 72.0}{100} = 59.0\%$$

The sucrose-to-water ratio (part sucrose to each part of water) in this (actual) syrup is

$$\left(\frac{S}{W}\right)_{\text{Act}} = \frac{S}{100 - DS} = \frac{59.0}{100 - 82.0} = 3.27$$

The sucrose-to-water ratio (sucrose solubility coefficient) at 60°C in syrup with a purity of 72.0% is 2.98 (taken from Table A.3), so K_{SS} is

$$K_{SS} = \frac{(S/W)_{\text{Act}}}{(S/W)_I} = \frac{3.27}{2.98} = 1.10$$

EXAMPLE 6

Given:

Low-raw (C) massecuite that has been in a cooling crystallizer for three days is centrifuged to produce molasses with the following analyses:

Temperature	45°C
Dry substance	81.0%
Sucrose content	48.6%
Purity	60.0

Calculate:

Supersaturation coefficient (K_{SS}) of molasses

$$S = \frac{DS \times P}{100} = \frac{81.0 \times 60.0}{100} = 48.6\%$$

$$K_{\text{Act}} = \left(\frac{S}{W}\right)_{\text{Act}} = \frac{S}{100 - DS} = \frac{48.6}{100 - 81.0} = 2.56$$

According to the Table A.3 (in the Appendix), K_I at 45°C in syrup with purity of 60.0% is 2.87, so K_{SS} will be

$$K_{SS} = \frac{K_{\text{Act}}}{K_I} = \frac{2.56}{2.87} = 0.89$$

This means that the molasses is undersaturated with sufficient sugar exhaustibility (K_{SS} = 0.89) during the evaporating crystallization as well as during the cooling crystallization. In other words, this is a satisfactory molasses.

SUPERSATURATION AND VISCOSITY, OPPOSING FORCES

In **sugar crystallization**, two forces act against each other: supersaturation (as the driving force) and viscosity (as the opposing force). Crystallization does not happen if the solution is not in a supersaturation situation (*the higher the supersaturation, the higher is the crystallization rate*). The factor that tends to inhibit the crystallization rate is the **viscosity** of the mother liquor (*the higher the viscosity, the lower is the crystallization rate*). Other factors such as temperature and stirring play important roles during crystallization as well, but once they are established and kept under control, their effects on optimization of the crystallization process become less important. Therefore, most of the discussion in this and the next station (Molasses Exhaustion) is limited to the establishment of the correct supersaturation (refers to K_{SS}) and *viscosity* in the mother liquor.

Viscosity is the measure of the resistance of a liquid to flow (*the higher the viscosity, the slower is the rate of flow*). Some liquids flow easily, some do not (water flows easily; whereas, honey does not). In principle, *the greater the attraction between molecules, the higher is the viscosity.* In a massecuite, viscosity is related to temperature (*T*), dry substance (*DS*), and purity of mother liquor. Molasses (the mother liquor of the final stage of crystallization) flows at a very slow rate and is the most *viscous liquid* in the sugar operation. This is because of its low purity (high nonsucroses) and high *DS* content. The crystallization rate (*R*) decreases when the mother liquor viscosity increases. The four important factors of the mother liquor influencing the *R* are:

- Supersaturation
- Dry substance
- Temperature
- Viscosity

Increasing supersaturation increases the *R* but increasing viscosity decreases or even stops the growth of crystals, particularly at the end of the final stage of the third crystallization. The role of viscosity in crystal growth is most pronounced when the purity of mother liquor lowers.

An increase in temperature decreases the viscosity and increases the *R* (*a temperature rise of 10°C in a syrup, doubles the R*). Figure 3.16.5 shows the changes in viscosity of a saturated pure sucrose solution in relation to temperature changes.

Increasing the dry substance of the mother liquor increases its viscosity. Figure 3.16.6 proves that viscosity of the molasses exponentially increases when its *DS* increases.

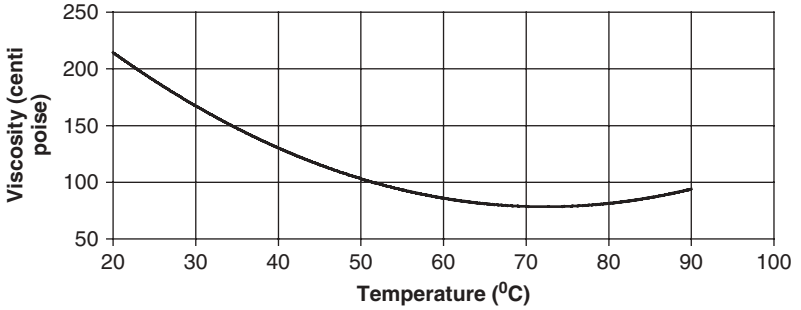


Figure 3.16.5 Effect of temperature on viscosity of saturated sucrose solutions

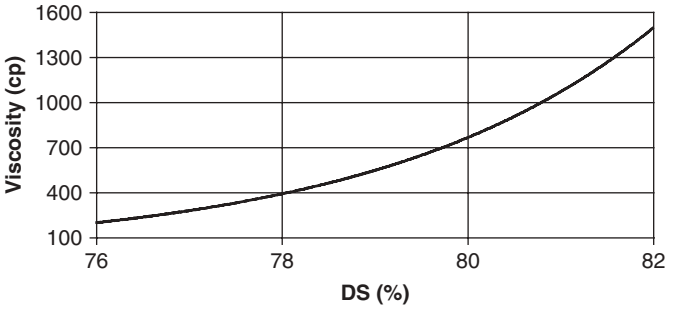


Figure 3.16.6 Effect of dry substance on viscosity in molasses with 60% purity at 50°C

NOTE

After nonsucroses, viscosity is the second most important parameter affecting sugar losses to molasses. Viscosity is important not only in sugar crystallization (particularly the low-raw side) but also in the transformation of in-process products in pipes, cooling crystallization, molasses exhaustion, and centrifuging of the massecuite. Viscosity is discussed more in the next section (Station 17).

CRYSTAL CONTENT

Crystal content (*CC*, or crystal yield) is the percentage (by mass) of crystals in the massecuite. *CC* can be calculated from the **purity drop** (purity difference) and from the difference in sucrose content in the massecuite and its mother liquor:

$$CC = \frac{DS_M(P_M - P_{ML})}{100 - P_{ML}} \tag{3.16.8}$$

$$CC = \frac{S_M - S_{ML}}{100 - S_M} \times 100 \tag{3.16.9}$$

DS_M	Dry substance of massecuite (%)
P_M	Purity of massecuite (%)
P_{ML}	Purity of mother liquor (%)
S_M	Sucrose content of massecuite (%)
S_{ML}	Sucrose content of mother liquor (%)

EXAMPLE 7

Given:

Dry substance of white massecuite	92.2%
Purity of white massecuite	92.2%
Purity of high-green (A) syrup	84.2%

Calculate:
Percentage of crystal content

$$CC = \frac{DS_M(P_M - P_{ML})}{100 - P_{ML}} = \frac{92.2\,(92.2 - 84.2)}{100 - 84.2} = 46.7\%$$

The values in the following table are calculated by using Eq. (3.16.8). They show the effect of massecuite dry substance (*DS*) on the crystal content for white and high-raw massecuite (Rhoten 2002). The values prove that a definite massecuite *DS* is required to achieve a definite crystal content in a massecuite at a given purity.

Effect of Mass *DS* on Crystal Content (*CC*)

Massecuite	*DS*	*CC*
White	91.0	50.2
White	91.5	52.3
High raw	92.0	46.2
High raw	92.5	47.4

Figure 3.16.7 shows the massecuite purity and *DS* relationship for the range of purity normally encountered in white and high-raw massecuite for an approximate 50% crystal content (*CC*). When the feed purity is low (viscosity is high), considerable time is required for the mother liquor to reach equilibrium and become saturated, resulting in a lower crystal content in the massecuite.

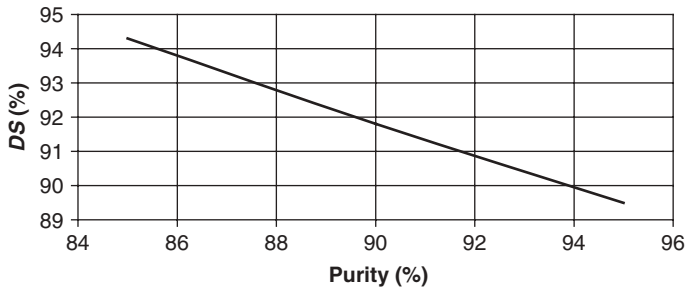


Figure 3.16.7 Effect of purity on massecuite dry substance at 50% *CC*

NOTE

Although high crystal content is desired, there is a limit. With higher crystal content, the massecuite becomes extremely *viscous* and loses mobility. This causes difficulties when discharging the massecuite from the crystallizer, mixing it in the mixer, and centrifuging. At discharge from the pan, white massecuite should have about 55% crystal content (CC), the high-raw massecuite about 45% CC, and the low-raw massecuite about 35% CC. The crystal content of the low-raw massecuite is increased (to about 36%) during cooling crystallization.

CRYSTALLIZATION IN A BEET-SUGAR FACTORY

Supersaturation (the driving force) must be established in a solution for crystallization to occur. You can form a supersaturated solution by the following methods:

- **Evaporation:** A saturated syrup becomes supersaturated by evaporation.
- **Flashing evaporation:** A saturated syrup becomes supersaturated by evaporation under vacuum.
- **Cooling:** The low-raw massecuite in the cooling crystallizer becomes supersaturated by a decrease in temperature (*sucrose solubility decreases when temperature decreases*).
- **Seeding:** The saturated-sucrose solution in the pan becomes supersaturated when the pan is seeded with fine sucrose particles.

In sugar factories, crystallization is performed by evaporation under vacuum for a number of reasons:

- **To reduce the boiling point** (*BP*)**:** The presence of the vacuum increases the temperature difference (ΔT) between steam and the boiling material, so the material boils at a lower temperature (*the higher the ΔT, the lower is the BP*). Toward the end of crystallization, the massecuite contains about 90% *DS*, which equals a boiling-point elevation (*BPE*) of about 20°C. This raises the *BP* to 120°C, which causes a high *color formation* if the process was not performed under vacuum.
- **To avoid inversion:** At low temperatures, the inversion of sucrose to invert sugar is minimal.
- **To save energy:** The energy is saved because of the lower *BP* of the syrup.

The vacuum in the crystallizer is maintained to correspond to a syrup boiling point of 75 to 80°C. The vacuum occurs by a condenser and a vacuum pump placed between the crystallizer and the condenser.

To recover the maximum amount of sucrose from thick juice and produce low-purity molasses, the sugar industry uses a three-stage crystallization process (refer to Figure 3.16.1). The stages are as follows:

- White crystallization (A-crystallization)
- High-raw crystallization (B-crystallization)
- Low-raw crystallization (C-crystallization)

There is a good reason why the sugar industry does not use a **one-stage** system to crystallize the thick juice and produce sugar. In a one-stage system, the crystallization would continue longer than usual to produce a **satisfactory molasses** (the molasses with purity of 60 or lower). Long crystallization time creates a massecuite with high concentration (above 96%) and high crystal content (above 70%). Such a massecuite would have extremely high viscosity. It would lose mobility, breaking the pan agitator and making centrifuging *impossible*. To prove this, let us calculate the crystal content of a massecuite produced from thick juice with a purity of 90 when the massecuite is concentrated to a high *DS* of 96% to produce a molasses with a purity of 60. Study the example.

EXAMPLE 8

Given:

Dry substance (*DS*) of white massecuite	96.0%
Purity of white massecuite (P_M)	90.0%
Purity of mother liquor (P_{ML})	60.0%

Calculate:

Percent crystal content (*CC*)

$$CC = \frac{DS_M(P_M - P_{ML})}{100 - P_{ML}} = \frac{96(90 - 60)}{100 - 60} = 72\%$$

This amount of crystal content in the massecuite creates an **impossible situation**. More crystals present in the massecuite means less mother liquor around the crystals, which stops the movement of the sucrose molecules from the mother liquor to the crystal structure.

There is a limit to the crystal content. Above 55% crystal content, the crystals are too close to each other, and the massecuite loses mobility and can even become so hard that it cannot be discharged from the pan. This is why the *CC* of white massecuite should not exceed 55%.

Purity drop (*PD* or **crystallization effect**) is a special term in crystallization operation. *PD* is defined as the difference in purity between the feed to the pan and the mother liquor centrifuged from the massecuite. In normal operations, standard liquor entering the sugar-end has a purity range of 90 to 92%. White massecuite is produced from the standard liquor, and it has the same purity (see the following Note). A **satisfactory molasses** should have a purity of approximately 60 or lower. This creates a *PD* in the range of about 30 to 32 units between the massecuite in the first stage (white massecuite) and the mother liquor of the third stage (molasses). In practice, such a *PD* is not achievable in one stage and must proceed step by step (normally in three stages). Generally, it is possible to achieve 6 to 8 *PD* in the first stage, 10 to 12 in the second, and 16 to 18 in the third, resulting in a satisfactory molasses with below 60% purity from the standard liquor with 90 to 92% purity.

NOTE

The purity (the percentage of sucrose in the total dry substance) of the massecuite is the same as the feed to the pan because *purity does not change with concentration, dilution, or crystallization*, assuming that no decomposition of sucrose occurs. In other words, during the evaporation, dilution, or crystallization process, the water is the only substance whose content changes, and purity is not relevant to water content.

As a help to understanding the crystallization process, the following is a review of three-stage crystallization (refer to Figure 3.16.1). (Note: The values are typical)

1. **Standard liquor** (with purity of about 93%) is boiled in the first crystallization stage (white pan) until it becomes supersaturated.
2. The supersaturated liquor is **seeded** and the crystals are given time to grow.
3. Boiling stops when the crystals reach the desired size. At this point, the product is called white (A) massecuite, which is discharged to the mixer.
4. The massecuite from the mixer is centrifuged in white (A) centrifuges to separate the crystals from the mother liquor. The sugar separated from the mother liquor (about 50% of the white massecuite is mother liquor) is white (A) sugar. The first runoff syrup that leaves the centrifuge before the addition of wash water is called **high-green** (A-green) syrup, which has about 86% purity.
5. The sugar crystals are washed again with a small amount of hot water (called **wash water**) during the second part of centrifugation. The syrup discharged during the wash (called **wash syrup**) has higher purity than high-green syrup because a small part of the crystals is dissolved during washing. Wash syrup is pumped to the melter.
6. The high-green syrup is collected in a tank and from there is pumped to high-raw (B) pan as feed for the second stage of crystallization.
7. The wash syrup is collected and pumped to the melter.
8. The high-green (A-green) syrup is seeded, and the crystals are given time to grow and produce high-raw (B) massecuite.
9. The high-raw massecuite is centrifuged in high-raw (B) centrifuges to produce high-raw (B) sugar and **low-green** (B-green) syrup, which has a purity of about 76%.
10. The high-raw sugar is sent to the melter.
11. The low-green (B-green) syrup is used to boil the low-raw (C) massecuite (third stage).
12. The low-raw massecuite is discharged to the mixer and then to the **cooling crystallizer**, where it stays for 2 to 3 days to complete the crystallization process.
13. After the holding period, the massecuite is centrifuged in the low-raw (C) centrifuges to produce low-raw (C) sugar and **molasses** (the mother liquor from the last stage).
14. The low-raw sugar is sent to the melter and the molasses is separated from the process to prevent the accumulation of impurities. The molasses is sold as a byproduct of the factory or desugared if the factory is equipped with **molasses-desugaring** equipment.
15. High-raw and low-raw sugars are melted by mixing them with thick juice and wash syrup in the melter to produce **standard liquor**, which is fed to the first crystallization stage.

EXAMPLE 9

Given:

Thick juice purity (P_J)	91.5%
Molasses purity (P_M)	60.0%

Calculate:
Recovery (R) of the sugar-end operations

Recovery is calculated by using recovery equation, Eq. (1.4), given in Section 2 of Chapter 1:

$$R = \frac{P_J - P_M}{P_J(100 - P_M)} \times 10000 = \frac{91.5 - 60.0}{91.5(100 - 60.0)} \times 10000 = 86.1\%$$

BATCH CRYSTALLIZATION

In a **batch-crystallization operation**, the sugar boiler performs the following steps to produce white massecuite: 1) preparation, 2) boiling, 3) seeding, 4) growing, 5) charging, 6) concentrating, 7) discharging, and 8) cleaning.

During crystallization, dry substance (DS) and temperature of the syrup, feed and steam flows, and steam pressure has to be maintained in order to establish proper curve between the crystallization zones. These include **stable zone** (the zone immediately below the saturation curve), **metastable zone** (the zone where crystal formation occurs by seeding), and **unstable or labile zone** (the unsafe zone). During the boiling stage (second step of crystallization), the syrup should be kept at the stable zone with supersaturation (refers to K_{SS}) slightly below 1 (saturated state) to prevent **nucleation** (the formation of crystals with or without seeding). When the syrup becomes concentrated and supersaturation passes 1, the metastable zone starts. At the beginning of this zone when K_{SS} is about 1.1, the seeding starts. To grow the crystals, the supersaturation is increased gradually, but it should never pass the safe zone (metastable zone), which corresponds to a supersaturation of above 1.6. In other words, from the time of seeding until the crystallization is complete, the massecuite should be kept at a different, but correct, supersaturation situation. There will be more discussion on of this later when we get to the crystallization steps.

Preparation

To prepare the pan for the new **strike** (cycle), the vacuum of the pan is adjusted by the condenser valve, so that the temperature of the boiling material can be maintained by vacuum pressure and the amount of steam. The **absolute pressure** is used to calibrate the vacuum pressure (absolute pressure = atmospheric pressure – vacuum pressure). For example, if the atmospheric pressure at the factory is 784 mm Hg (31 in. Hg) and the crystallizer is under 574 mm (22.6 in.) vacuum, the absolute pressure is 210 mm Hg (8.3 in. mercury). If the vacuum remains constant, the absolute pressure changes with a change in atmospheric pressure. In the sugar industry, an instrument holds the absolute pressure constant to maintain the vacuum regardless of the atmospheric pressure, which depends on the geographic location of the factory.

At this point, we should remember that the crystallization process should be conducted under steady pressure in order to simplify the control supersaturation. For white crystallization, an absolute pressure of 210 to 240 mm Hg (8.3 to 9.5 in.) is usually recommended because boiling below 210 mm can contribute to false-grain formation. On the other hand, absolute pressure above 240 mm raises the massecuite temperature, causing an increase in color. If the vacuum cannot be kept steady, the pan temperature will be influenced by the pressure of the steam chest. As a result, the sugar boiler will not be able to control the evaporation rate. If the vacuum is under control, the control of the evaporation rate becomes easy (in scientific terms, the control of supersaturation becomes easy) because the process can be controlled by the following easily controlled variables:

- Feed rate
- Steam rate
- Temperature

In the following section, we will see how the sugar boiler keeps these variables under close control during each step of crystallization.

Boiling

Boiling is the second stage of crystallization during which the syrup is concentrated under vacuum (at about 74°C) to the desired *DS* and supersaturation for seeding. Boiling is performed by the vapor supplied from the evaporation station. As the vacuum begins to rise, the syrup valve is opened, and the first charge of hot undersaturated feed is fed into the pan. The **belt height** (the level of the feed surface in the pan) is usually maintained to the point where it covers the heating surface of the pan (usually 25% of the total useful volume of the pan). Then the charge valve is closed, and the steam valve is opened to evaporate the syrup. The smaller feed valve is opened enough to keep the heating surface just covered. A slower boiling is maintained and then gradually increased until supersaturation is reached. The rate of steam depends on the factory's vapor-distribution system. White and high-raw pans usually take vapor from the third-effect evaporators. In any case, sugar boilers avoid a fast boiling because it can cause **entrainment** (carrying boiling material droplets by the vapor into the condenser). At a given vacuum, the temperature of the syrup increases steadily as it continues to evaporate and becomes more concentrated. (In an average white-pan boiling, about 15 t of water are evaporated every hour.)

Seeding

Seeding is performed at the appropriate temperature and concentration (scientifically, at the right supersaturation) of the syrup. Syrup concentration and temperature are good indications of supersaturation (refers to K_{SS}) because of their close relation. Syrup concentration and temperature are easy to control by ordinary instruments At 210 mm (8.3 in.) absolute pressure, when the temperature reaches about 74°C, a typical syrup with a purity of about 92 has the right K_{SS} (1.1 to 1.2) to be seeded. At this point, seed is applied by opening the seed valve to seed the pan (seed preparation is described later).

For seeding, the temperature must be carefully controlled because the temperature increases considerably when the syrup is concentrated and enters the metastable zone. This

makes the seeding time easier and more accurate because of the sensitivity of K_{SS} to temperature (*a 4°C change in syrup temperature result in a change of about 0.4* K_{SS}). After seeding, the pan boiler uses the pan microscope to watch the growth of the crystals because the seed particles become visible immediately after seeding.

The white pan should be seeded at supersaturation of 1.1 to 1.2 with the proper amount of seed slurry (described later). When the pan is not automated, a **proof test** (putting a small amount of syrup between two fingers and then moving the fingers apart until the syrup breaks) is used.

Sugar boilers choose the right seeding time also by observing the bubbles and the splash on the sight glasses at the syrup level. The bubbles rise slowly, and the splashes run down slowly, as the syrup becomes more concentrated and ready for seeding.

The proof test and observation of the bubbles and splashes give sugar boilers feeling for viscosity of the syrup; they can estimate the concentration and supersaturation of the syrup because viscosity is related to concentration and therefore to supersaturation. In the proof test, and to the trained eye of a sugar boiler, for example, a short string (about 2.5 cm or 1 in.) corresponds to the right time for seeding. A longer string means the correct time for seeding has passed. Seeding at low supersaturation (K_{SS} below 1.1) causes some crystals to dissolve (because the syrup is still in the stable zone), leaving fewer particles to grow. On the other hand, seeding at a high supersaturation (K_{SS} above 1.3 in white pan) creates the following problems:

- High viscosity
- Unwanted false crystals
- Unwanted conglomerates

Later, you will learn about instruments that automatically determine the parameters affecting the crystallization, particularly concentration, which can indirectly indicate the state of supersaturation (refers to supersaturation coefficient, K_{SS}) of the syrup.

Growing

The **growing of crystals** is the movement of sucrose molecules from the solution phase to the crystal (solid) phase. Supersaturation is the driving force for this movement (crystals do not grow if K_{SS} is not maintained). The subject of crystal growth is complex, so it will be covered in more detail later. However, it should be clear at this point that the supersaturation should be watched carefully while the crystals are growing.

After seeding, the crystals are tiny and far apart, so the sugar boiler lets them grow further to the desired size. At a constant vacuum, the massecuite temperature is a good guide for keeping the mother liquor in the intermediate zone by gradual evaporation and for adding more feed. In other words, *as the syrup concentration increases, the feed rate must increase to maintain the same level of supersaturation*. The important point is to avoid too large a supersaturation because of **false-grain formation** (the formation of small unwanted crystals). This is actually a **recrystallization** proces, in which a new set of grains is formed. Too low a supersaturation dissolves the crystals. If the massecuite under crystallization has a high purity (e.g., white-boiling with over 90 purity), it usually takes a short time (5 to 10 minutes) for crystals to grow to size. At this time, the crystals have sharp faces and are closer to each other. This is easily seen using the pan's microscope or by touching the crystals on a proof glass (see Figure 3.16.8).



Figure 3.16.8 Watching massecuite on a proof glass

Charging

Charging is adding more feed to the pan. At the charging stage, the pan gets more charge and steam is gradually increased to keep the massecuite in a steady supersaturated situation. Charging is a critical stage because feed, which is undersaturated, may dissolve the crystals. Therefore, the supersaturation should be kept in a safe zone by close control of the massecuite temperature. To prevent this, the steam and feed intakes are regulated to keep the temperature slightly below 76°C. If the temperature passes the safe value, the crystals can **conglomerate** (the connection of two or more crystals).

As the normal pan volume is approached, the feed rate is reduced and finally closed. At this point, the crystals look sharp and uniform with almost no conglomerate and no false crystals.

Remember, you can do two things to keep the temperature of the massecuite in the safe limit (slightly lower than 76°C):

- Increase the feed rate
- Decrease the steam flow

The first remedy is the best if there is room in the pan. If not, try both remedies moderately. In general, when the white pan is at a proper feed rate, the massecuite temperature may fluctuate one degree without serious difficulty. However, keep the temperature at half degree below the upper safe limit.

Concentrating

Concentrating (tightening) is stopping to feed, letting the crystals grow to the desired size, and concentrating the massecuite to the desired *DS*. The white pan is boiled first in the range

of 25 to 32% crystal content (*CC*) and concentrated to about 93% *DS* to achieve a final CC of 50 to 55%.

Keeping a proper rate of crystal growth by maintaining the right supersaturation (K_{SS} of about 1.5) is the most important point at this stage. The concentrating of white massecuite takes 30 to 90 minutes, depending on the purity of the massecuite. The concentration and supersaturation of the massecuite increases as the crystals grow.

In a normal-size pan with about 50 m^3 (1770 ft^3) useful capacity, about one ton of sugar moves from the syrup to the crystal state. If the supersaturation is not suitable, this sugar does not set on the surface of existing crystals but produces small crystals of its own. The small crystals cannot then reach a usable size in the time remaining, and they create difficulties, such as the following:

- Decreasing crystallization efficiency
- Creating difficulty during centrifuging
- Creating crystals with insufficient uniformity
- Increasing mother-liquor purity during centrifugation (fine crystals pass centrifugal screen and go to the green syrup)

Unsatisfactory work at this stage means that the sugar boiler has to boil the syrup again later in the day. A low yield of sugar increases the quantity of the syrup that has to be boiled later in the raw pans (high-raw and low-raw pans). For this reason, it is important to control closely the concentration of the massecuite and its supersaturation values. To keep the temperature below 76°C and supersaturation below the false-grain level (K_{SS} below 1.6), the steam input should be regulated and enough time should be given to finish the job correctly. Any effort to push the process by using a high evaporation rate will decrease the crystallization efficiency and uniformity of crystals.

Discharging

The end of the concentrating step is determined by the increasing stiffness of the proof test and the slow movement of bubbles across the glass. At the right massecuite concentration (white massecuite can be concentrated to 92 to 93% *DS*), the steam is closed and the vacuum-breaker valve is opened. As the vacuum drops to almost half its value, the discharge valve is opened to discharge the massecuite into the mixer.

Cleaning

After the massecuite is discharged, the discharge valve is closed and the drain valve is opened to clean the pan. The pan is cleaned by steam injection and sometimes also by a hot water spray. The pan should be thoroughly cleaned to avoid uncontrolled nucleation during the next strike. The wash water usually is pumped to the melter or centrifugal receiving tank. In preparation for the next strike, the steam, vacuum-breaker, discharge, and drain valves are closed, and the vacuum is turned on.

Here, let us follow the crystallization steps of standard liquor on a graph. As shown in Figure 3.16.9, the entire area to the right of the saturation curve ($K_{SS} = 1$) represents an undersaturated (stable) zone, and the area to the left of the curve represents the supersaturated (metastable) zone. The steps in Figure 3.16.9 proceed as follow:

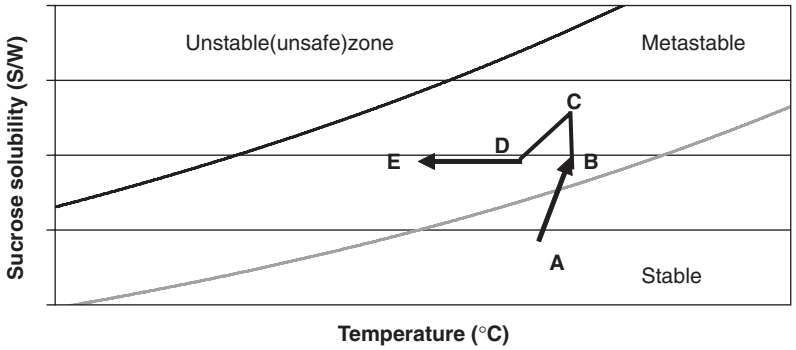


Figure 3.16.9 Crystallization patterns

1. **Precharging and evaporation:** First, the liquor is undersaturated (point A). During boiling, the liquor follows line AB until it becomes supersaturated.
2. **Seeding:** At about midstage of supersaturation (K_{SS} of about 1.15), seed is added to the liquor at point B and crystallization starts.
3. **Growing:** Supersaturation is maintained around point C by gradual evaporation and by adding more feed at a constant temperature.
4. **Charging:** The large feed is introduced, which reduces the temperature and concentration, causing a mild reduction of supersaturation to point D.
5. **Concentrating:** The supersaturation is kept in the safe zone (metastable zone) by steady evaporation until the desired concentration is achieved at point E, at which time the pan is ready for discharge.

Following are the typical time spans for the individual steps of white pan crystallization:

Boiling	25 minutes
Building	60 minutes
Concentrating	25 minutes
Total	110 minutes

The **building time** consists of the seeding, growing, and charging period, so the majority of the total boiling time is spent to build the pan. (The boiling times of the individual stages of crystallization differ.) The time for the first (white) crystallization is about 2 hours, that of the second crystallization is 4 to 8 hours, and the third crystallization takes 8 to 12 hours. The difference in processing time is mainly due to the differences in purity of the syrup under crystallization (*the lower the purity, the lower is the crystallization rate and the longer is the crystallization time*). In general, low purity (high nonsucrose content) of the syrup.

- Decreases the mobility of the molecules
- Decreases the crystallization rate
- Increases viscosity of the massecuite

CRYSTALLIZATION CONTROL AND AUTOMATION

Sugar crystallization is a delicate process and needs to be controlled by either a skilled sugar boiler or an automated control system. When sugar crystallization is operator-controlled,

the results are dependent on the skill of the sugar boiler. Critical points such as seeding time, concentration, and supersaturation coefficient (K_{SS}) are estimated by observations and results from laboratory tests such as the purity test on the massecuite and the mother liquor. The observations are not reliable (no two sugar boilers control the crystallization in the same way), and the laboratory results are usually after-the-fact information (ready after completion of the strike).

To make the sugar boiler's job more reliable, sugar technologists and programmers have tried to automate the crystallization operation by measuring the critical parameters on line, and using integrated control systems and computer programs. John Ziegler (employee of Taylor Instrument, U.S.A., for 36 years until 1972) was one of the pioneers in the field of crystallization automation (Blickley 1991).

Implementing an automatic control system with programmed sequences is a way to handle variations in feed and vapor supply, vacuum, temperature of feed, vapor, and condensate, and speed of the pan stirrer to achieve efficient crystallization. The crystallization is usually operated from the pan-floor control room by the sugar boiler, who has access to sufficient, accurate data.

Linking lab data into the automatic control system and displaying the data on the computer in the pan-floor control room can eliminate after-the-fact problems in lab results. The linking system continuously displays lab data on the computer screen and updates the data as the lab analysts enter the results of tests. Critical control variables can be displayed graphically with upper and lower control ranges. With this system, the sugar boiler can make the necessary adjustments to keep the process in the desired range. It is recommended that each data point be displayed as an average of several lab analyses. The averaging method reduces the variations in data. Figure 3.16.10 shows an example of a sugar-end purity profile displayed in the form of a chart on the computer.

As stated, controlling supersaturation (refers to the supersaturation coefficient, K_{SS}) is the most important point in the crystallization process. But what makes the automation job difficult is that supersaturation *cannot be measured directly*. Sadly, sugar scientists have not yet developed an instrument to reliably measure K_{SS} directly. Therefore, other variables must be measured by different techniques, calculated, and related to supersaturation. This is a time-consuming process. Although the calculations are no longer a problem because they are handled by computer programs, relating different parameters to K_{SS} affects the **accuracy** of the measurements. In addition, the data tables on K_{SS} are not suitable for a computer-control system. Therefore,

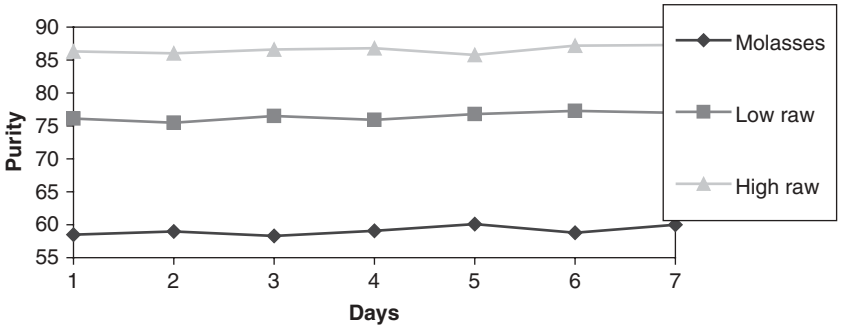


Figure 3.16.10 Sugar-end purity profile

without automated systems that can handle the entire crystallization process sufficiently, skilled sugar boilers must share their knowledge with the control system to run the crystallization station efficiently, as is explained next.

The four main interrelated variables affecting the supersaturation coefficient (K_{SS}) during crystallization are the mother-liquor dry substance (DS_{ML}), purity (P_{ML}), temperature (T_{ML}), and saturation coefficient (K_{IP}). Note that *ML* subscript indicates **mother liquor**. The K_{SS} can be expressed as a function of the four independent variables as follows (Rozsa 1996):

$$K_{SS} = f(DS_{ML} P_{ML} T_{ML} K_{IP}) \tag{3.16.10}$$

Each of these variables relates to supersaturation (ss) in different ways as follows:

- **Dry substance:** The mother-liquor concentration has the largest effect on *SS*, making it the most important parameter to measure during crystallization. The larger the *DS*, the larger is the *SS* and the larger is the sucrose content. This means that the relation between *DS* and K_{SS} is *proportional*. With a high *DS* feed, the limitation in sugar crystallization shifts from an evaporation rate to a crystallization rate. The evaporation rate must be reduced to avoid a high supersaturation and unwanted secondary (false) crystals.
- **Purity:** During concentration, the mother-liquor purity does not change. It is equal to the feed purity, which is usually known from lab tests. When the syrup is ready to be seeded, the *DS* and temperature increase considerably, which helps determine *SS* and controls the seeding time. But after seeding, the purity decreases gradually because of sucrose movement from the mother liquor to the crystals. The supersaturation increases considerably as the purity of the mother liquor decreases when concentration is kept constant. This creates a *nonproportional (*irrelevant) *relation* between purity and supersaturation.
- **Temperature:** An increase in temperature results in an increase in supersaturation (a proportional relation), and this applies through the entire process. In white crystallization, a 4°C change in pan temperature changes supersaturation by about 0.4 units. In high-raw and low-raw crystallization, 4 to 8°C change creates the same change in supersaturation. As stated before, a change in the pan vacuum changes the temperature of the massecuite. If the vacuum cannot be controlled, the pan temperature will also be influenced by the steam pressure in the steam chest, reducing the sugar boiler's ability to control supersaturation in the syrup. Remember that the only way to control supersaturation is to control the temperature of the massecuite.
- **Saturation coefficient** (K_{IP}): Mother liquors with different K_{IP} can hold different amounts of sucrose, resulting in different K_{SS}. This is because of the difference in nonsucroses characteristics, which mainly depends on the characteristics of the beets.

The preceding can help a *pan programmer* understand how supersaturation coefficient (K_{SS}) can be controlled by the different mother-liquor parameters. To control crystallization (actually, to determine K_{SS}), a number of techniques are used in the sugar industry:

- Refractometric method
- Microwave method
- Temperature-controlled method
- Boiling-point elevation method

- Conductometric method
- Dielectric-constant method
- Viscosity method

Refractometric Method

The **refractometric method** is based on the direct relation between the concentration and the **refractive index** (RI) of a syrup at a constant temperature. This way, RI can indirectly represent the concentration (dry substance) of the sample. Special refractometers are available for online measurement of concentration. Unlike a lab refractometer, the online refractometers are not affected by the presence of crystals in the massecuite. These refractometers are equipped with an *energy source* such as *cesium*, which is capable of giving electromagnetic energy by gamma rays with a long useful duration (50% of the capacity is lost in 30 years). The energy source is placed in a double-wall, waterproof, stainless steel box, which is mounted in the bottom of the pan. The source sends constant energy to the *detector*, which is installed outside the pan. The amount of energy reaching the detector is inversely proportional to the massecuite concentration. The concentration values enable the control of the supersaturation of the syrup under the crystallization process. These refractometers are also equipped with automatic temperature compensation (ATP) for variations in the standard temperature, which is 20°C.

Microwave Method

The **microwave method** uses a microwave probe to measure the water content of a syrup or massecuite. The dry substance can be determined by direct measurement of the water content. This method is a reliable and cost-effective way for controlling the crystallization process.

Microwaves have a longer wavelength than infrared (IR). Microwaves have special energy to rotate the molecules, and the heat produced by the rotating molecules can quickly evaporate moisture content.

Temperature-Controlled Method

The **temperature-controlled method** is based on the direct relationship between solubility and temperature (*sucrose solubility increases appreciably with temperature*). At a given temperature, any increase in concentration influences the equilibrium and creates a different supersaturation condition.

At a constant absolute pressure, temperature is directly related to the supersaturation coefficient (K_{SS}). Therefore, K_{SS} can be expressed as a temperature difference instead of a concentration difference. Figure 3.16.11 shows the relation between temperature and saturation of a pure solution. In addition to the solubility curve ($K_{SS} = 1.0$), the figure shows a K_{SS} curve of 1.3. The entire area to the right of the solubility curve represents undersaturation, and the area to the left of this curve represents supersaturation.

Boiling-Point-Elevation Method

The **boiling point elevation** (*BPE*) of a solution is the solution's boiling point above that of pure water. The normal boiling point of a solution is the temperature at which its vapor

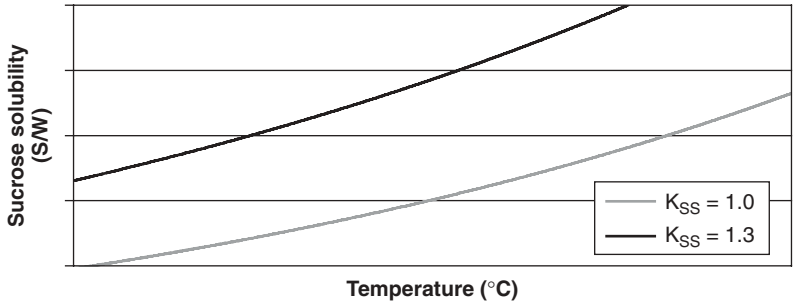


Figure 3.16.11 Effect of temperature on sucrose solubility

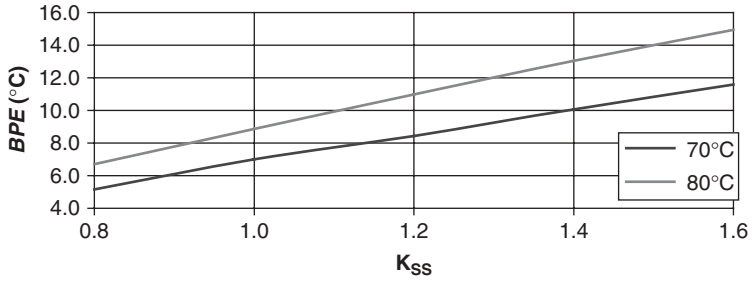


Figure 3.16.12 Relation between *BPE* and supersaturation coefficient at 70 and 80°C

pressure is equal to atmospheric pressure (1 atm = 1.013 bar = 760 mm Hg [torr] = 14.7 psi). At normal atmospheric pressure (sea level), syrups increase the boiling point of water to above 100°C. For example, sugar solution with 65% dry substance boils at 103.8°C (refer to Table A.4 in the Appendix). The same syrup boils at 83.3°C under vacuum corresponding to a boiling point of the water of 80°C (similar to the situation in a vacuum pan).

During crystallization, *BPE* occurs because of a change in the concentration and supersaturation coefficient (K_{SS}) of solutes (sucrose and nonsucrose substances) (see Figure 3.16.12). As the dry substance of a solution increases, its *BPE* increases as well because the presence of solute increases the boiling point of the solutions. The *BPE* method is based on the fact that *the BPE of the syrup is related proportionally to its dry-substance content (concentration) and is higher than that of pure water.* Therefore, the concentration can be indirectly determined by temperature, which represents *BPE*.

In the *BPE* method, the temperature of the material under boiling and in the vapor space of the pan (or absolute pressure) are measured and used to determine the concentration (dry substance) from which supersaturation can be estimated. This method is used infrequently in the sugar industry because *BPE* is affected by vapor-pressure variations, causing deviations in the signals used for control. In addition, the accuracy of the method depends on the devices measuring the temperature.

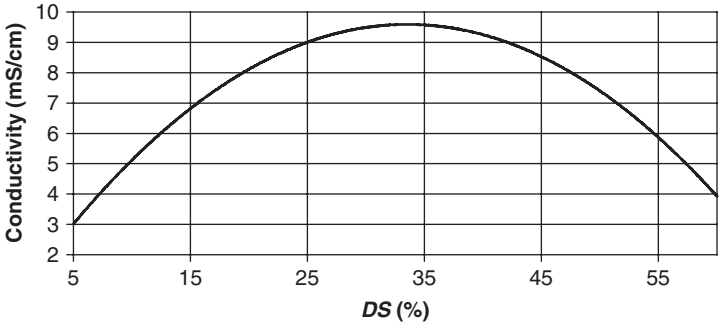


Figure 3.16.13 Relation between conductivity and dry substance of standard liquor at 60°C

Conductometric Method

The conductometric method (conductivity method) is based on the resistance of electric current carried out through a solution. *The conductivity of the syrup decreases with increased concentration*. As Figure 3.16.13 shows, impure sucrose solutions with dry-substance contents between 25 and 30% have the largest conductivity. Each solute has its own degree of conductance, so the measure of solution conductance is proportionally related to the concentration of the substance, from which supersaturation can be estimated.

Conductivity increases with increasing temperature and *decreases with increasing purity* because of low ash content in high-purity syrups. The following table compares the specific conductivity (K) of some in-process products:

	Purity	K (mS/cm)
Standard liquor	93	6.5
A-green syrup	85	14.0
B-green syrup	76	22.0
Molasses	60	36.5

Conductivity increases with increasing nonsucroses (decreasing purity). Suspended solids and precipitated sediments do not affect conductivity (Bubnik et al. 1995). Although the conductivity method requires keeping several variables constant (e.g., feed *DS*, temperature, and vapor pressure), it correlates more closely with the supersaturation coefficient (K_{SS}) of the syrup than with its concentration. *The conductivity of the massecuite decreases with increasing K_{SS} and with increasing crystal content*. Figure 3.16.14 shows the change in conductivity during white crystallization.

New conductivity probes (e.g., Fletcher Smith) have no electrical contact with the massecuite but measure conductivity by sensing the dielectric loss using radio frequency. This provides greater sensitivity and reduces signal drift. However, conductivity method cannot be used successfully in high-purity massecuite (above 80% purity).

Viscosity Method

The **viscosity method** is based on the direct relation between the viscosity, concentration, and supersaturation coefficient (*viscosity changes noticeably with concentration and therefore with K_{SS}*).

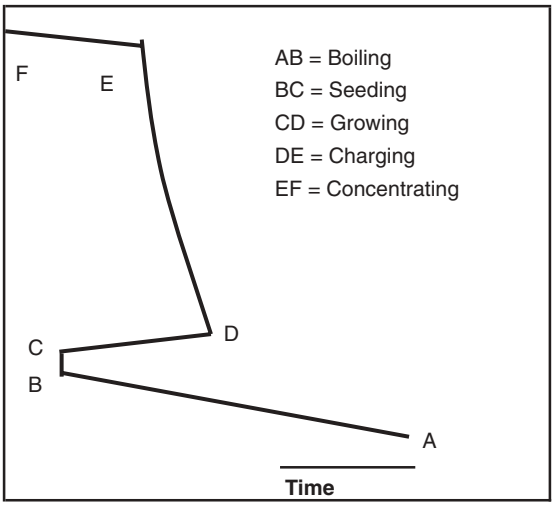


Figure 3.16.14 Conductivity patterns during white crystallization

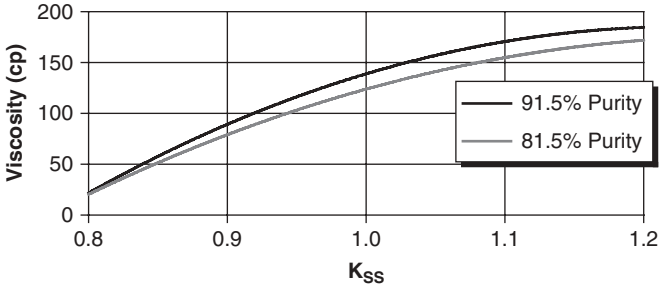


Figure 3.16.15 Relationship between viscosity and K_{SS} of impure sucrose solutions

For a given temperature, the supersaturation coefficient (K_{SS}) of the mother liquor can be identified by its viscosity (Figure 3.16.15), particularly during seeding.

Some industrial viscosimeters consist of an electrically driven rotating device. The torque (turning moment) of the rotating device is proportional to the current. The current values can be related to viscosity and indirectly to the supersaturation of the syrup. The viscosimeter can be mounted on the pan wall. The values measured by the viscosimeter works well up to the point where crystals in the mother liquor are not grown. Toward the end of crystallization (as the crystals are larger), the measurement values do not reflect the exact viscosity of the syrup.

SUGAR BOILER

The **sugar boiler** is the person in charge of operation of the crystallization process (the most interesting process in the factory). Beet-sugar factories usually have one sugar boiler per shift to operate the white pans and one to operate the **raw-side pans** (high-raw and low-raw pans). The boiler in charge of the raw side is called the **raw-sugar boiler**. Sugar boilers, whether in

charge of white or raw pans, must have both practical and theoretical knowledge to operate the crystallization station efficiently. In the sugar boiler's language, an *experienced sugar boiler boils the syrup on the first pass, an inexperienced one boils the syrup again later in the day*. An unskilled sugar boiler produces a massecuite with such fine crystals that they easily pass the centrifuge screen and become part of the mother liquor, which has to come back to the boiler again for crystallization.

Sugar boilers should be familiar not only with mechanics of the job but also with the fundamentals of sugar crystallization and instruments used in the crystallization station. They should know how to take full advantage of all factors that optimize the sugar-crystallization process. In addition, today's sugar boiler must be a **computer user**.

The sugar boiler should be familiar with the following subjects:

- The effect of the crystallization process on energy usage
- How crystal content (*CC*) affects crystallization efficiency
- Making uniform crystals (low *CV*) and the right size of crystals (right *MA*)
- The emergency responses necessary for steam, feed, and vacuum failure
- How molasses production with low purity affects the efficiency of the factory

Information such as mean aperture (*MA*), coefficient of variation (*CV*), crystal content (*CC*), and crystallization efficiency is usually included in the daily laboratory report, which should be reviewed by the sugar boiler.

Sugar boilers or their supervisors *should never force the pan operation*. Interest in **doing the right job** to produce high-quality sugar should always be the practice in the crystallization operation. A good job on the raw side contributes to the quality of remelt sugar and the production of low-purity molasses.

NOTE

There is no difference between the two classes of sugar boilers (white and raw side); both should know the basics of the sugar-crystallization process.

SEED PREPARATION

Seeding is the process of adding fine particles (that function as a base for crystal growth to a supersaturated liquor.) Each seed particle is the base for a single crystal that grows to a desired size by absorbing sucrose from the mother liquor around it.

Seed characteristics affect the crystallization yield and the uniformity of the sucrose crystals as follows:

- **Seed particle size:** The size of the seed particles affects the **crystallization rate** (*R*). *R* is directly proportional to the crystal surface, so crystallization is faster when the size of the seed is smaller and their numbers are larger because the total surface is then greater. In addition, when finer seed is used, the risk of false-grain formation is lower. The difference between small and large particles is that the small ones create a large surface energy per unit weight, which has a positive effect on crystal growth.
- **Seed uniformity:** Small crystals can more easily stay in equilibrium condition with a supersaturated solution than larger ones. If large crystals are also present in the solution, the

equilibrium cannot last because the smaller crystals will dissolve and only the larger ones will grow. This type of crystallization produces an undesirable low **crystal content** (the percentage of crystals in the massecuite).

Small (5 to 10 micron), uniform seed particles produce the best result. During crystallization, these small particles grow to a desired size of 350 to 600 µm (*MA* = 350 to 600), depending on the customer's demands.

Pans are seeded with:

- Powdered sugar
- Slurry (fondant)
- Seed magma
- Massecuite

Seeding with fine powdered sugar is performed by adding a certain amount of powdered sugar to the pan at the time of seeding. The amount of seed is about 200 g (half pounds) per each 50 m^3 (1770 ft^3) of syrup. The size of the powdered-sugar particles should be about 10 microns. (Note: This type of seeding is not widely used because of the difficulty in controlling the size and uniformity of the powdered-sugar particles.)

Seeding with slurry (fondant) offers better control of the size and uniformity of the seed particles. The slurry is made by wet **milling** (grounding) granulated-refined sugar in isopropyl alcohol ($CH_3CHOHCH_3$) in a small ball mill with a porcelain container (e.g., SWECO vibratory ball mill). A standard procedure must be followed to produce a slurry that is consistent in solid content, the number of crystals per unit volume, and the mean crystal size. In some factories, 2.3 kg (5 lb) of sugar are mixed with 5 liters of isopropyl alcohol and milled for 5 hours. In the United States, the procedure can be called the **triple-five process**, which is based on 5 lb of sugar, 5 L of alcohol, and 5 hours of grounding. The size of the particles in the produced slurry is usually 5 to 10 microns (µm) and each mL of slurry contains about 2.5×10^9 particles. For a medium to large pan, 500 mL of the slurry are used in the first-stage crystallization and 300 mL in the second and third stages of crystallization. This amount of seed is required for fine crystal boiling. With coarse crystals, the pan needs less seed.

A company in Europe offers a commercial slurry, under the trade name Seed Crystal Fondant, that has a fine particle size (about 7 µm) with high uniformity.

Seeding with seed magma is produced by mixing low-raw (C) sugar with green syrup (mostly low-green syrup) to produce seed magma (see the following Notes) for high-raw (B) boiling. This method is mainly used for seeding continuous crystallizers in high-raw (B) boiling.

Seeding with massecuite is produced by batch boiling and used in some factories for seeding continuous crystallizers in low-raw (C) crystallization.

NOTES

- **The mixture of seed crystals and syrup is usually called seed magma to distinguish magma from massecuite, because both terms refer to a mixture of crystals and syrup. Massecuite *is the product of the pan, and seed magma is seed that is used in the pan.***
- **The purity of seed magma is important because the adjustment of the final massecuite purity is largely achieved by adjusting the seed-magma purity.**

CRYSTALLIZATION THEORY

So far, you have learned the practical aspects of the crystallization process. In addition, you learned how to compare undersaturated, saturated, and supersaturated solutions as well as the effects of impurities on sucrose solubility. But you still need to know what happens during crystallization in the pan.

We will start from the time when the syrup under crystallization is undersaturated ($K_{SS} < 1$). To make the subject easier, we will consider a system containing a *pure sucrose solution* and ignore the presence of nonsucroses because they have an insignificant effect on sucrose solubility. For example, the sucrose solubility of a pure sugar syrup at 90°C is 4.2 and that in a 90 purity syrup is 4.1 g per g water.

At low concentrations (undersaturated situation), the syrup contains many free water molecules, so water can easily dissolve the sucrose (sugar) molecules. As evaporation continues, the number of free water molecules and water molecules bonded to each sucrose molecule is gradually reduced until the syrup becomes saturated ($K_{SS} = 1$). *At saturation, the system is in equilibrium*. When the system reaches the saturation point, the properties of the syrup change; for example, the concentration and viscosity of the syrup increases. When the syrup is seeded, a *crystalline-slurry system* is formed, and the system gets to a supersaturated condition ($K_{SS} > 1$). This condition is not stable because it is not at equilibrium. Again, *saturation is an equilibrium mechanism and supersaturation is not*. Therefore, a supersaturated solution repeatedly changes its condition accordingly until equilibrium reestablishes.

As the evaporation brings the crystals closer together, the equilibrium condition disturbs the crystals, causing the excess sucrose in the syrup to be absorbed by the crystals until a saturated solution is formed again. With an increase in the concentration of the sucrose in the solution by the continuation of evaporation, the stress on the equilibrium again shifts the syrup to a supersaturated situation to absorb this stress. Only by the *shift of equilibrium* can the crystals absorb more sucrose and reduce the stress. The equilibrium shifts because of the concentration difference between the syrup, in which the crystals are growing, and the saturated syrup, which is on each crystal's surface and is in equilibrium with the crystals. This is the basis of the general statement: *the concentration difference in a supersaturated solution is the driving force that causes the crystals to grow.* (A positive difference causes sucrose crystals to grow, and a negative difference dissolves them.)

Crystallization is a two-step process:

- Nucleation
- Crystal growth

Nucleation is the formation of crystals with or without **seeding** at a given supersaturation coefficient (K_{SS}). An equilibrium condition is reached when the solution is saturated at a given temperature, which can be shown by the solubility curve to be a function of temperature. Figure 3.16.16 helps elucidate the nucleation process. For example, let us consider point *A*, from which a supersaturated condition can be obtained by evaporation (1), by flashing evaporation (2), or by cooling (3).

From Figure 3.16.16, the crystallization zones can be defined as:

- **Stable** (saturated): In this area, neither nucleation nor growth can happen
- **Metastable** (safe): In this area, nucleation can happen only in the presence of seed
- **Unstable** (unsafe): In this area, nucleation can happen in the presence or absence of seed

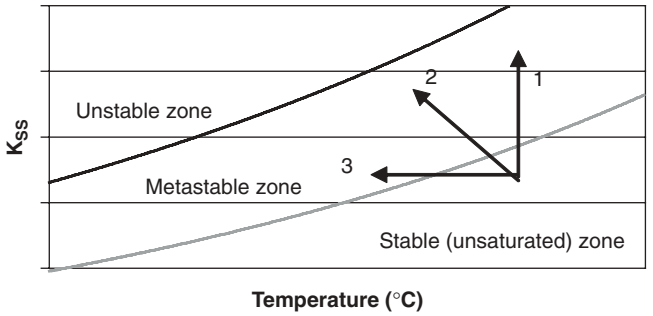


Figure 3.16.16 Creation of supersaturation by an increase or decrease in temperature

(Note: Both the metastable and unstable zones are in supersaturated positions.) At low supersaturation ($K_{SS} < 1.1$), which is in the metastable zone, no new crystals are created, and only existing crystals gradually grow. On the other hand, we know that *the higher the* supersaturation, *the faster the crystals grow*. Therefore, the supersaturation should be increased, but there is a limit to it because supersaturation should be kept below the upper safety zone ($K_{SS} < 1.6$) to prevent the creation of new (false) crystals.

Seeding is one of the most important steps in the crystallization operation because it determines the size and yield of the crystals in the final product. The following should be taken into consideration during the seeding process:

- Ensure that the seed is at the right size particles (5 to 10 μm)
- Ensure that the seeding is performed at the right K_{SS} (in white pan 1.1 to 1.2)

In the rest of this discussion, the causes of **crystal growth** (the solidification of molecules from a solution phase) are considered. (Note: Sugar technologists, who wants to know the details of the crystallization process must study the complex subject of crystal growth so that they can understand the factors that affect the rate at which crystallization proceeds.)

Crystal growth is expressed as a **crystallization rate** (R_C), which is the amount of the sucrose crystallized in 1 minute on $1\,m^2$ of the crystal surface. Crystal growth is a two-step process. The first step, which occurs in the syrup (the mother liquor), is a mass (weight) transfer by the **diffusion process**, in which the sucrose molecules from the solution phase are transferred to the crystal's surface. The second step, which occurs at the crystal's surface, is a position change by the **surface reaction**, in which the sucrose molecules position themselves into the crystal's structure. (Note: There must be enough heat energy to allow individual molecules to move around, position themselves on exiting crystals, and make stable bonds with them.)

To understand what happens in the second step, we should look at the **adsorption-layer theory** (Montovani and Vaccari 1998). When a sucrose molecule (see Figure 3.16.17) reaches the crystal surface, it starts to create a stable bond with the existing crystal structure. The possibility of positioning the sucrose molecule into the crystal structure depends on the bonding between the molecules, which happens when supersaturation and heat energy exist. If the bonding does not happen (in the absence of supersaturation and heat energy), the sucrose molecule goes back again to the solution phase. In the preaence of SS and heat energy, more sucrose molecules join the crystal structure and fill the empty spaces, making the crystal faces flat and complete.

The driving force of the first step (the diffusion process) is directly proportional to the concentration difference between the diffusion layer (C) and the crystal surface (C_1). The driving force for the second step (the surface reaction) is the concentration difference between the crystal surface and the adsorption layer (C_0). Note that equilibrium exists at the interface between the liquid and solid (crystal) phases. In other words, the syrup concentration on the adsorption layer (C_0) corresponds to a saturated syrup, but at a distance from the crystal surface, the syrup is supersaturated and its concentration is C. First, the sucrose molecule diffuses through the distance of d_1 and when it approaches the crystal, immediately sits on it; therefore, C_0 remains the same. After eliminating the unknown C_1 and combining the diffusion equation of both steps, the crystallization rate (R_C) for a unit base [mass (m) per time (t)] can be expressed by the following equation:

$$\text{Crystal growth} = R_C = \frac{dm}{dt} = K_D(C - C_0) \tag{3.16.11}$$

The equation takes the surface area of all crystals (A) to express the overall growth:

$$R_C = K_D \times A(C - C_0) \tag{3.16.12}$$

C	Sucrose concentration in the mother liquor
C_0	Sucrose concentration on the crystal surface
K_D	Diffusion coefficient
A	Overall surface area

The K_D is the **diffusion coefficient** (the overall mass-transfer coefficient), defined as the rate of mass transfer per unit area per unit concentration difference. The concentrations can be expressed in mass percent (g/100 g water) or mole fractions. According to Einstein (1879–1955), the K_D depends on the temperature of the syrup (T) and its viscosity (η):

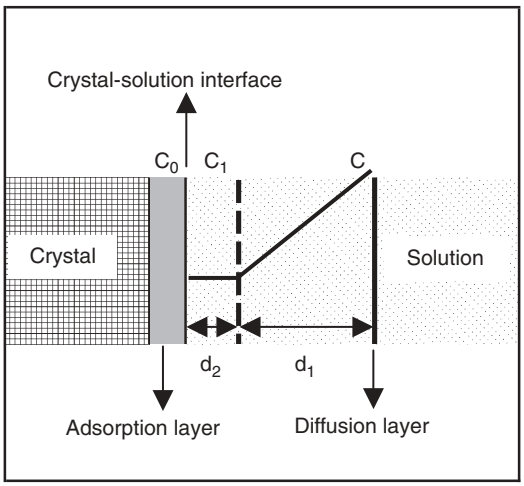


Figure 3.16.17 Crystal growth

$$K_D = \frac{KT}{\eta} \tag{3.16.13}$$

K Constant, dependent on the molecular mass of solute (in our case, sucrose)

Substituting K_D from Eq. (3.16.13) into (3.16.12) yields

$$R_C = \frac{KT}{\eta A}(C - C_0) \tag{3.16.14}$$

Equation (3.16.14) represents both steps together. This means that the sucrose molecule moves (by diffusion) at a rate of R_C from the solution with viscosity of η to the crystal with surface of A and continues (by surface reaction) at the same rate to establish itself into the crystal structure.

If the crystallization were dependent on only the first step (diffusion), all the crystals would have a round shape. The presence of the second step (surface reaction) completes the shaping of the crystal. The factors affecting the crystallization rate (R_C) are as follows:

- **Size of seed grain:** R_C increases (sugar crystallizes faster) in massecuite with fine crystals because they have a larger total crystal area.
- **Temperature** (T): R_C increases with increasing T (see Figure 3.16.18).
- **Purity of syrup** (P): R_C increases with increasing P (see Figure 3.16.19). In other words, the presence of impurities slows crystal growth. For example, first stage (white) crystallization

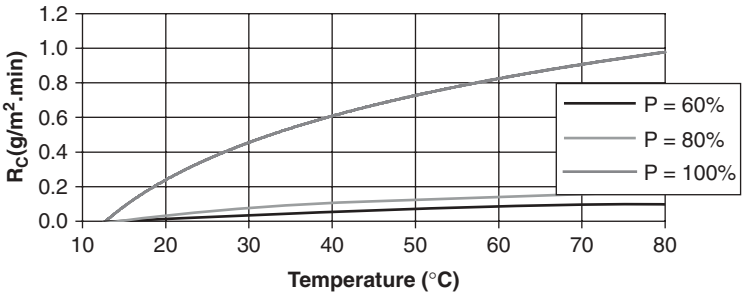


Figure 3.16.18 Relation between R_C and temperature

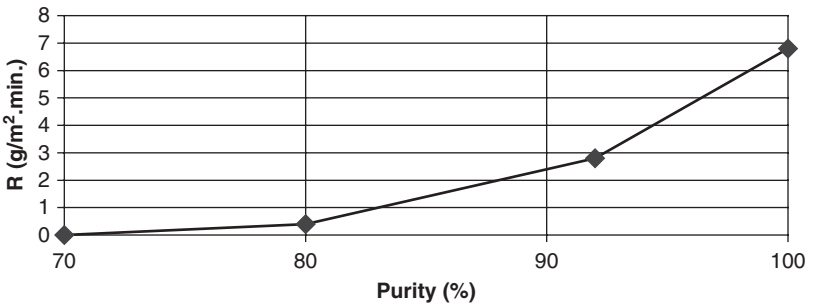


Figure 3.16.19 Relation between R_C and purity

usually takes about 2 hours; the second stage takes about 5 hours; and the third stage takes 8 to 12 hours, depending on the purity of the syrup.

- **Supersaturation coefficient** (K_{SS}): R_C increases with increasing K_{SS} (see 3.16.20). Low-purity syrups take longer to reach a supersaturation situation (the more the solution is supersaturated, the higher is its R_C).
- **Viscosity:** R_C decreases with increasing viscosity.
- **Stirring:** R_C increases with stirring (see Figure 3.16.21).

In general, **crystal growth** occurs layer by layer on the crystal face and the growth rate of crystals follows McCabe's rule (McCabe et al. 2001), which states that *all crystals grow at the same rate if the K_{SS} stays the same for all crystals*. Therefore, the crystals in a massecuite with an K_{SS} of 1.10 grow three times faster than that in a massecuite with an K_{SS} of 1.03:

$$\frac{1.10-1.00}{1.03-1.00}=\frac{0.10}{0.03}=3$$

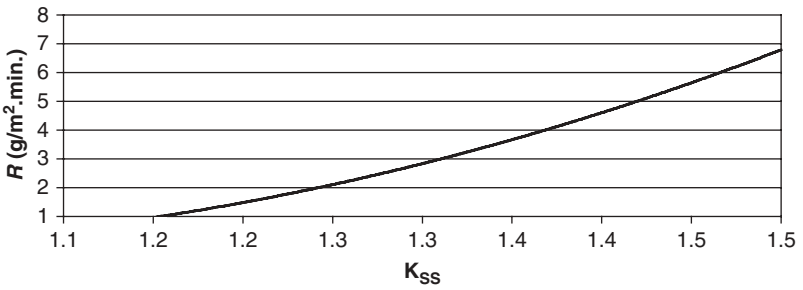


Figure 3.16.20 Relation between R_C and K_{SS}

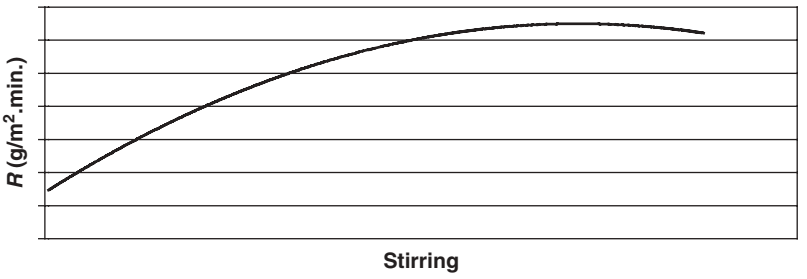


Figure 3.16.21 Relation between R_C and stirring

NOTES

- **When a crystal forms, heat (energy) is released from the solution, known as the heat of crystallization. This is the reason why crystallization is considered an exothermic process (a releasing-energy reaction).**
- **At a typical temperature of 75°C, sucrose solubility is 3.5, meaning 1 kg water contains 3.5 kg of sugar. Therefore, for 1 kg of water evaporated from the massecuite, about 3.5 kg of sugar is transferred from the syrup to the crystals.**
- **An important point in the crystallization process is the transformation of colorants from the syrup to the crystal structure. The subject of color transformation to the crystal structure is complicated, and much depends on the type and nature of the colorants in the syrup. The difference in the nature of colorants is evident when the final beet syrup for crystallization (standard liquor) is compared with the final cane syrup. A standard liquor of 2000 to 3000 IU (CUMSA unit) color can produce a 20 to 30 IU granulated-refined sugar, whereas cane syrup must be in the 200 to 300 IU range to produce sugar with similar color.**

CRYSTALLOGRAPHY OF SUCROSE

Crystallography is the science of studying the structure and characteristics of crystals at their atomic or molecular level. The particles of crystalline solids are arranged in a systematic-geometric pattern, called a **crystal lattice**. Each crystalline solid has its own three-dimensionally shaped lattice with surfaces (called **faces** in crystallography) at specific angles (called **interfacial angles**) to each other. Crystals are classified based on the interfacial angles as follows: monoclinic, triclinic, cubic, hexagonal, trigonal, tetragonal, and orthorhombic. A given substance may crystallize in two or more types, depending on the crystallization conditions.

Sucrose crystallizes in a monoclinic shape (see Figure 3.16.22) with up to 15 faces, depending on impurities in the syrup and the growing conditions. Because molecules of a sucrose crystal are **nonionic** (see Section 4 in Chapter 1), they are arranged in a regular order that allow light to pass through the crystal. Therefore, sucrose crystals look transparent.

The formation of **twin crystals** (two joined single crystals) is due to the accidental conglomeration of two crystals that grow together during the crystallization process. According to Montovani (1995), the following can contribute to the formation of twin crystals:

- Crystallization under high supersaturation
- Seed characteristics
- Syrup purity
- Stirring

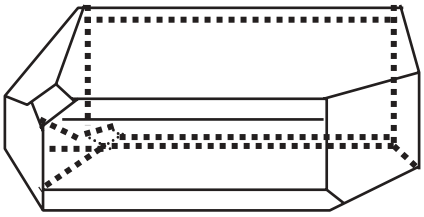


Figure 3.16.22 A sucrose crystal grown in a pure sucrose solution

Impurities in sugar-end syrups have a big effect on the crystal's shape. Crystals produced from syrup with a high raffinose (a trisaccharide) content have increased **elongation** along the B- and C-axes, which can be seen by the naked eye when the crystals are large. Dextran (a polysaccharide) is one of the impurities that affects crystallization as a *growth inhibitor* and *deforms the crystal*. In general, the crystals produced from a syrup containing a high content of raffinose, invert sucrose, or polysaccharides are different in shape. The crystal grows further if a **conglomerate** (two or more crystals together) of molecules grows, resulting in the increase of some sites of the crystal. This situation occurs when rock candy is produced.

CRYSTALLIZERS

The **crystallizer** (crystallizer pan) is a vessel with auxiliaries (e.g., feeding and discharge equipment) for the crystallization process. Two types of crystallizers are used in sugar plants:

- Evaporating crystallizers
- Cooling crystallizers (explained in next section–Station 17)

Evaporating crystallizers are of two types:

- Batch crystallizers
- Continuous crystallizers

Batch Evaporating Crystallizers

In its **batch** design, an evaporating **crystallizer** (crystallizer pan), as shown in Figure 3.16.23, is similar to an evaporator. It is a cylindrical vessel with an almost equal height and diameter.

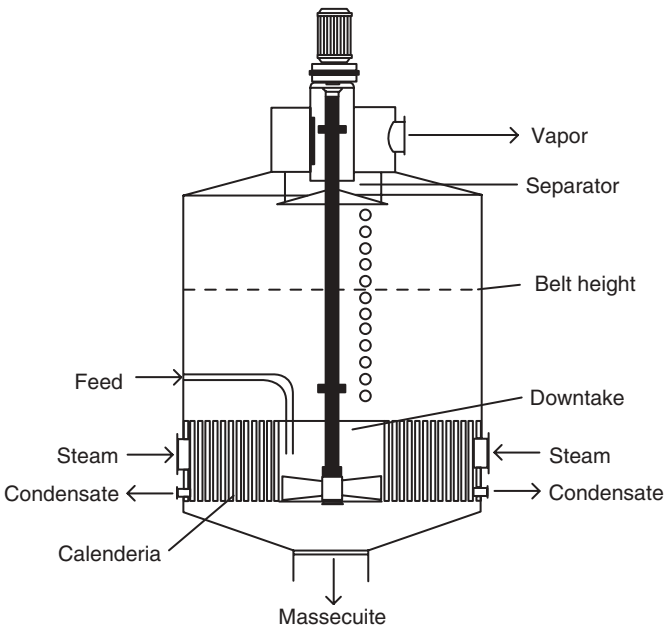


Figure 3.16.23 Batch crystallizer with a top-drive stirrer

Vacuum pans are made with capacity from 40 to 100 t massecuite. For example, a pan 5 m (15) high and 4.5 m (13.5 ft) in diameter has a volume of 80 m^3 (2820 ft^3). (Note: The actual height of the massecuite at the end of crystallization is held at about 2 m (6 ft) above the upper plate of the steam chest to ensure the elimination of carrying over boiling material to the condenser.) The level of the boiling surface is called the **belt height**. This makes the useful volume of about 50 m^3 (1770 ft^3), which is approximately 60% of the total volume of the pan. With consideration of the massecuite density of about 1.5 t/m^3, the useful capacity of such a crystallizer is about 75 t. A condenser and a pump installed between the condenser and the crystallizer produce the vacuum supplied to the crystallizer.

The **calandria** (heating tubes and a steam chest) is filled with vertical heating tubes with an inside diameter of 80 to 120 mm (3 to 4.7 in.) Some evaporators have stainless-steel tubes 100 mm in diameter and 1.5 mm thick. Some crystallizer suppliers offer hexagonal tubes, which have the advantage of having a larger surface area than round tubes.

The heating tubes are rolled into the holes of the **tube sheets** (two perforated sheets, placed horizontally at a distance of 2 to 3 m from each other). The heating tubes (see Figure 3.16.24) are rolled into the holes of the tube sheets. In general, pan tubes (see Figure 3.16.24) are larger than evaporator tubes to circulate the massecuite more easily. A wide downtake (30 to 50% of the crystallizer diameter) is fitted into the center of the chest.

Steam with about 1 atm (14.7 psi), usually supplied by the second, third, or fourth vapor from the evaporators, is introduced into the steam chest of the pan to heat the tubes. Depending on the crystallizer size, the height of the tubes is from 2 to 4 m (6 to 12 ft). An entrainment separator at the top of the evaporator removes any boiling-material droplets carried by the vapor. Condensate is removed from the bottom of the chest by two condensate outlets. The vapor leaves the crystallizer through a vacuum valve to the condenser. **Noncon-**

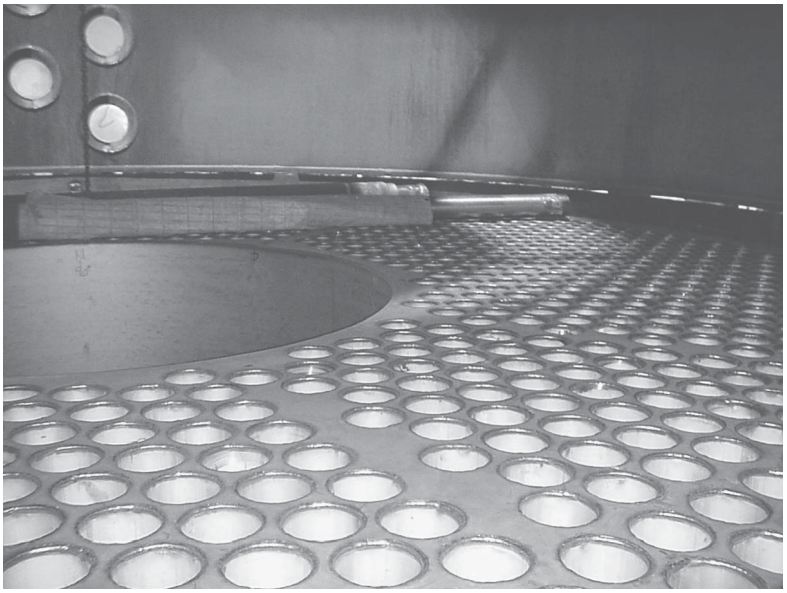


Figure 3.16.24 Crystallizer tubes

densable gasses (e.g., air, ammonia, and carbon dioxide) are discharged from the bottom of the calandria.

A good **stirring** of the boiling material is important in crystallization to equalize the concentration and the temperature of the massecuite. The boiling material is stirred by an impeller-type stirrer. The stirrer is driven from the top of the pan, and its impeller turns in the downtake tube to mix the massecuite. The impeller diameter is close to the inside of the downtake tube. Impellers are driven from the top or bottom of the crystallizers by a shaft, connected to an electric motor. The top-driven stirrers have a suspended shaft and are the most common. The direction of rotation is usually chosen to achieve a good circulation. In general, the volume of the boiling material stirred by the impeller must be great enough to circulate the entire material in a reasonable time. Due to the high viscosity of massecuite, an impeller with a strong motor is used.

The discharge outlet is large with a discharge valve that usually works hydraulically. Usually, a few sight glasses, on the body of the crystallizer, are available for observing the boiling material. For taking samples, a sampling cock is installed in the lower part. The cock is specially designed to not draw air into the crystallizer. Some pans are equipped with a simple refractometer to measure the concentration of the boiling syrup.

A special microscope on the side of the crystallizer magnifies the crystals so that the sugar boiler can better control the boiling operation. Steam injectors inside the crystallizer are used to clean it after each strike. A pressure gauge is also fitted to indicate the vacuum of the upper part of the crystallizer. A thermometer is installed above the calandria, and another one at full-pan level to show the temperature of the boiling material.

Continuous Evaporating Crystallizers

In the 1980s, beet-sugar factories started to use the new type of evaporating crystallizer, the **continuous evaporating crystallizer**. Today, some beet-sugar factories around the world are equipped with continuous crystallizers for all three stages of crystallization or for raw-side products. Continuous crystallizers have the following advantages over batch types:

- Higher efficiency
- Easier process control
- Higher energy efficiency
- Less floor space required
- Choice of installation outside the main processing building
- More-for-less system (usually one crystallizer instead of four)

Their main disadvantages are as follows:

- More cleaning (boil-out) of scale deposits
- Less uniform crystals (lower coefficient of variation, *CV*)
- Difficulty with boiling syrup with purity above 97 (not suitable for refineries)

Continuous crystallizers are of horizontal or vertical designs. Figure 3.16.25 shows the sectional design of a **Fletcher Smith** (FS) continuous horizontal crystallizer. In the FS crystallizer, low-pressure steam heats the syrup in a plate-type heating element. The FS continuous pans are used for white crystallization. Good circulation allows the pan to be operated with as low as a

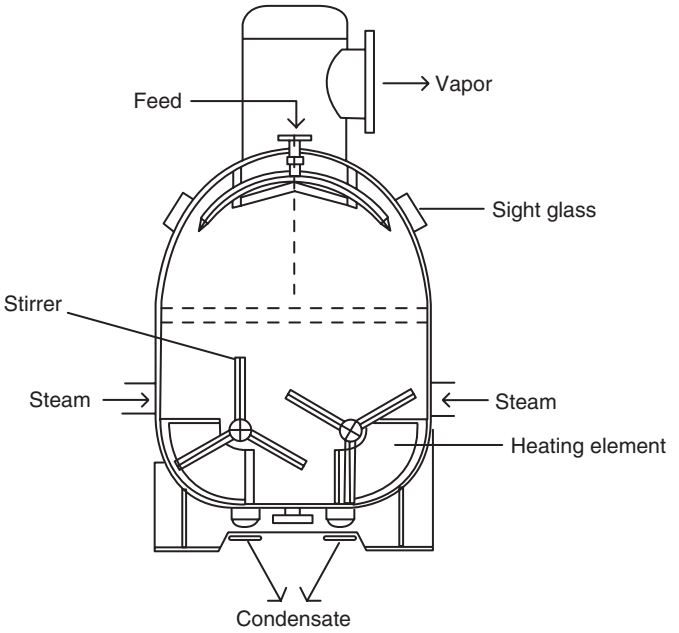


Figure 3.16.25 FS continuous horizontal crystallizer

15°C temperature difference between the steam and syrup. This is an efficient feature for factories with vapor compression. The heating elements are mechanically wiped by the stirrers, creating a high heat-transfer coefficient. The massecuite circulates down the center and up along the sidewalls. The massecuite is circulated by two counter-rotating stirrers, which requires less cleaning.

The **BMA** continuous vertical crystallizer, called **VKT**, consists of four or five chambers (compartments) installed on top of each other. The five-chamber model is used in larger factories, but the four-chamber model is the most common. The VKT (see Figure 3.16.26) consists of a cylindrical tank separated into four chambers that each look likes a batch crystallizer. The chambers are separated, and each has an impeller for mixing. Impellers are driven from the top of the compartment by a shaft connected to a motor. In addition, each chamber contains its own vapor line, calendria, and fittings (parts attached to equipment, such as valves and measuring devices). The heating tubes are made of stainless steel. The heating tubes are welded. The feed and the seed magma enter the top chamber and move by gravity to the chamber below. Finally, the massecuite leaves from the bottom of the last chamber.

VKT crystallizers are energy efficient because they use low-pressure steam. The low-pressure steam requirement led to the use of vapor from the second and third boiling in white VKT for the first time in the Gustrow sugar factory (van der Poel et al. 1998). The separation of chambers allows them to be bypassed for cleaning. During **cleaning**, the content of the dirty chamber is emptied into the chamber below, and a procedure similar to that for evaporator cleaning is followed. The continuous crystallizer for white boiling should be cleaned about every 15 days. For low-raw crystallization, chambers 1 and 2 should be cleaned every 45 days, chambers 3 and 4 might not need cleaning during the campaign (Van der Poel et al. 1998).

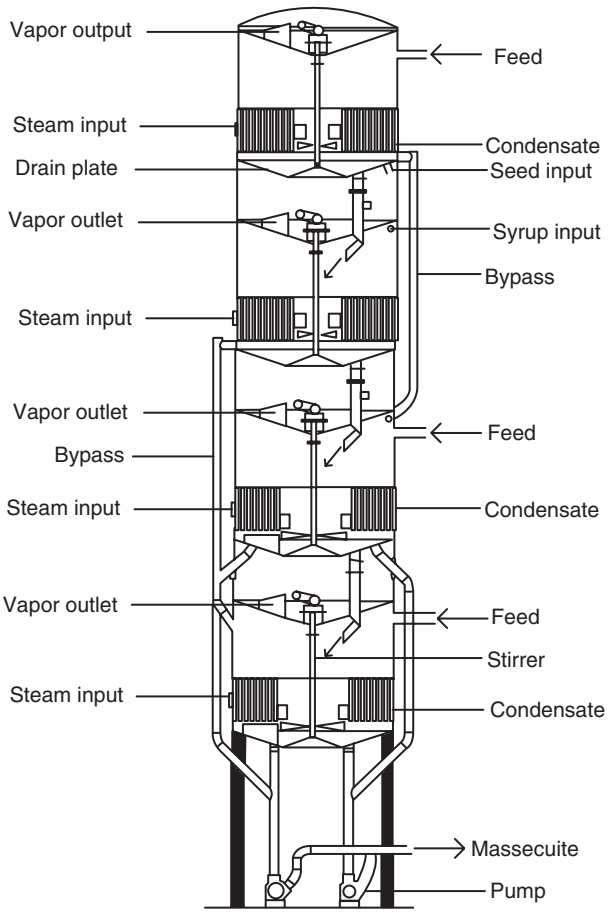


Figure 3.16.26 BMA continuous vertical crystallizer

When operating the VKT, the following three subjects should be controlled:

- Feed-to-seed ratio
- Dry substance in individual chambers
- Massecuite purity

For more information about optimum conditions for the operation of the VKT, refer to the next section (Station 18).

OPERATING PROBLEMS

A number of **problems** can occur in the crystallization operations.

Low Vacuum in the Pan

The condenser maintains the vacuum in the pan. The crystallization process is performed under uniform vacuum. For crystallization, the sugar boiler needs an absolute pressure of about

240 mm Hg (9.5 in.). At 780 atmospheric pressure, this equates to 540 mm (780 – 240 = 540.). Low vacuum occurs because of one or more of the following:

- **High temperature of cooling water in the condenser:** This happens usually at the beginning of the campaign when the weather is still warm (late September or early October).
- **Low amount of cooling water in the condenser**
- **Vacuum pump not functioning properly**
- **Leak in the condenser fittings**

Sugar in Condenser Water

Sugar in the **condenser water** (mixture of condensate and cooling water produced by a condenser) should not exceed 0.02%. Sugar **carrying over from the pan** can be the main cause for high sugar in condenser water. The height of massecuite in the pan should not be held more than 2 m (6 ft) above the upper plate of the steam chest to ensure the elimination of carrying over boiling material to the condenser.

Low Crystallization Rate

The normal boiling times for the crystallization stages are about 2 hours for the first-stage crystallization, 4 to 8 hours for the second stage, and 8 to 12 hours for the third stage. The case differences are because of the differences in the purity of the syrup under crystallization. Slow crystallization indicates the malfunctioning of one or more factors affecting crystallization. Possible causes of low crystallization rate are as follows:

- **Insufficient amount of steam:** Steam for crystallization is usually supplied by the second, third, or fourth vapor from evaporators with about 1 atm (14.7 psi) pressure. Low-value steam causes slow boiling.
- **Insufficient exit of condensate:** Sometimes part of the steam chest is filled with condensate, releasing less steam to the chest and lowering the crystallization rate. Therefore, the condensate outlet should be examined when the boiling starts to slow.
- **Wrong pH profile:** A pH drop across the sugar-end and its effect on crystallization is important (see Station 10, Juice Purification). The pH condition for growing crystals should be at a normal level, which is different for different stages. The optimum pH is 8.2 to 8.4 for white massecuite, 7.8 to 8.0 for the high-raw operation, and 7.4 to 7.6 for low-raw operation. With this pH profile, a slightly alkaline molasses (7.2 to 7.4 pH) is produced. If the pH profile is low, caustic is added to the standard liquor. The high pH of standard liquor can be adjusted with 3% hydrochloric acid to achieve the desired pH.

 A high pH profile causes the formation of saccharates, which slow the crystallization rate and increase massecuite viscosity and molasses purity considerably. On the white side, crystallization does not proceed as rapidly as usual when the pH is above 8.5. If the natural alkalinity is too high (this is either when beets contain high natural alkalinity or an excessive amount of caustic is used), called **high-buffered juice**, you will notice an unusual high pH profile and slow crystallization behavior throughout the sugar-end. When the sugar-end operation starts with a high pH standard liquor, the pH drop throughout the crystallization stages becomes abnormally low. The reason for the low pH drop is that the solutions with a higher pH become more basic when they are concentrated. On the other hand, if the

natural alkalinity of the juice is low, called **low-buffered juice**, the pH drop across the sugar-end operation is more than usual.

- **Insufficient stirring in the pan:** Insufficient stirring in the crystallizer causes crystals stay at the bottom of the pan, so there is little crystal surface exposed to the supersaturated solution present at the higher area.
- **Insufficient exit of noncondensable gasses**
- **Excessive scale on heating tubes**
- **Insufficient heating surface**
- **Low feed concentration**

Dead Boiling

Dead boiling is a term used when the crystallization at the start of boiling proceeds at a normal rate but starts to slow down although there is no fluctuation in the steam input to the pan. Following are some remedies when dead boiling happens:

- Mainly when the alkalinity of the syrup is low, although sometimes high alkalinity causes dead boiling as well. If alkalinity of the feed is low (i.e. addition of a few drops of the phenolphthalein to the feed sample does not change the color of the sample), caustic (NaOH) should be added to the pan. If alkalinity is high, 3% hydrochloric acid can be mixed with the feed in small doses.
- **Surfactant** (a surface-active substance that reduces the surface tension of solutions) can be added to the pan.
- Charge the pan with different syrups.
- Decrease the temperature of the diffusion process since one of the main reasons of dead boiling is the presence of pectin in the feed. This happens when the diffuser is operated at higher than normal temperatures (above 73°C). High temperature ruptures beet cells, causing the passing of **pectin** into the juice.

CHAPTER 3

SECTION 17

MOLASSES EXHAUSTION

SUBSECTIONS

- Molasses Formation
- Normal and Satisfactory Molasses
- Damage of High-Purity Molasses
- Production of Molasses with Satisfactory Purity
- High-Raw and Low-Raw Crystallization
- Affination
- Molasses Recycle
- Cooling Crystallization
- Cooling Crystallizers
- Massecuite Reheating before Centrifuging
- Operating Problems

ABOUT THIS SECTION **This section** gives information on **Station 17** (Molasses Exhaustion) of sugarbeet processing. Station 17 (see Figure 3.17.1) includes the low-raw side (second and third stages of crystallization) and cooling crystallization equipment. The process of remelting high-raw (B) and low-raw (C) sugar in the thick juice to produce standard liquor is also included in this station.

In a conventional sugarbeet operation, the amount of sugar lost to molasses is the largest loss (about 80% of unrecoverable sugar ends up in molasses). Therefore, maximum **molasses exhaustibility** (leaving the least amount of sugar in molasses) is the key to efficiency, and, consequently, to profitability of any beet-sugar factory. The easiest way to judge molasses exhaustibility is to evaluate the **purity** of molasses (*the lower the purity of molasses, the lower is the amount of sugar lost to molasses and, consequently, the higher is the sugar production*). Simply speaking, producing molasses with lowest possible purity is better, and a credit to the sugar factory, than producing molasses with high

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purity, which is the worst mistake that a factory can make. Therefore, the purity of molasses is important data recorded in the daily report of operation.

Sugar technologists need to understand how to operate the crystallization process (particularly, the raw-side operation) to produce molasses with the lowest possible purity without reducing the capacity of the factory. In sugar technology, the molasses with the lowest possible purity is called **exhausted molasses** (molasses with the lowest purity), so the process is known as molasses exhaustion. Molasses purity is affected by both the quality of the sugarbeet and the factory's equipment, so it is difficult to give an absolute definition for exhausted molasses. However, a general term, **satisfactory molasses**, can be **applied**.

In this section, raw-sugar boilers and sugar technologists will learn how to operate the raw side (high-raw and low-raw) of the crystallization operation. Most of the discussion will be directed toward the high-raw (B), low-raw (C), and cooling crystallization for optimization of the molasses exhaustibility.

Most important, you will learn how to produce molasses with satisfactory purity, *independently* of the purity of the standard liquor, according to three rules:

- **Rule 1, purity drop:** Try to achieve the right **purity drop** (PD) in each crystallization stage (the higher the PD, the more sugar crystallization in the pan and the lower is the purity of the mother liquor throughout the crystallization stages and, consequently, the lower is the molasses purity).
- **Rule 2, backboiling:** Try to achieve the right high-raw (B) massecuite purity (83 to 85%), independently of the standard liquor purity, by backboiling (the lower the purity of the starting target-point, the lower is the purity of the mother liquor and, consequently, the lower is the molasses purity).
- **Rule 3, massecuite concentration:** Try to achieve the right massecuite concentration (93.5 to 95.0% *DS* for high-raw and 94.5 to 96.0% *DS* for low-raw massecuite) at all stages (the higher the massecuite concentration, the lower is the purity of the mother liquor and, consequently, the lower is the molasses purity).

Exhaustion is the removal of a valuable substance from a compound to the maximum extent possible. **Sugar** (sucrose) is the most valuable substance present in sugarbeet, and its place at the final stage of sugar production should be in the bag (or silo), not in the molasses. In sugar technology, the action of reducing molasses purity to the lowest possible level (to a satisfactory level) is called **molasses exhaustion**.

Every sugar factory makes molasses. In the crystallization process, the sucrose-to-nonsucrose (*S/NS*) ratio reaches a point where the further crystallization of such low-purity syrup is almost impossible. This syrup, called **molasses**, must be separated from the process to prevent **nonsucrose** (nonsugar) accumulation.

In sugar technology, sugar in molasses is considered sugar loss. Reducing the sugar loss in molasses is one of the most important goals of the sugar factory because it increases profitability. The easiest way to evaluate the performance of a sugar factory is molasses purity (*the lower the molasses purity, the less sugar is left in the molasses, at the same amount of molasses production*). Production of molasses with low purity is the best credit that a beet factory can get. Thus, exhaustibility of molasses is the most important subject in running a beet facility.

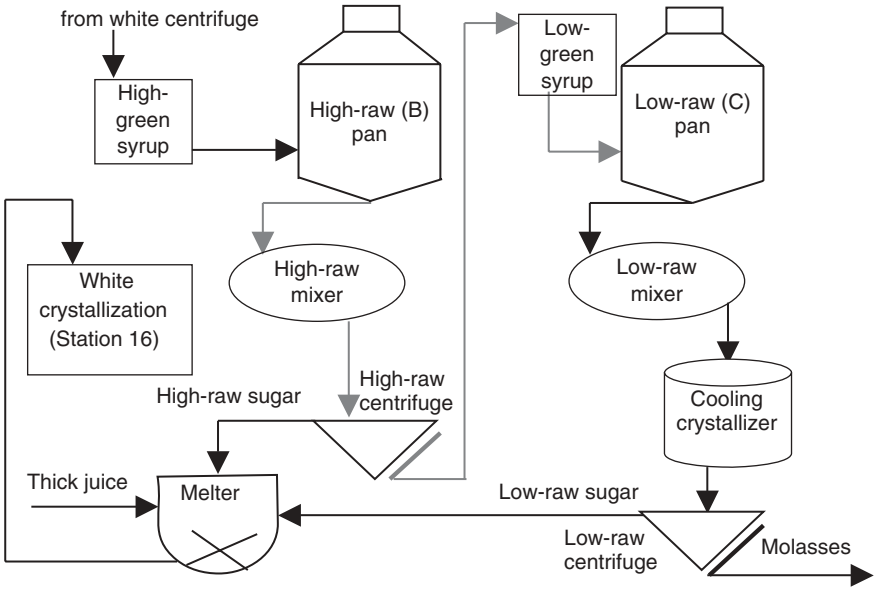


Figure 3.17.1 Flow diagram of raw-side crystallization (Station 17)

The goals of the raw-side (the second and third stages) crystallization are to produce

- Molasses with low purity
- Raw sugar with low color
- Raw sugar with high purity
- Raw sugar with uniform crystals
- Raw sugar with easy centrifugibility

Molasses is usually produced at about 4 to 5% on beet (OB). It must not contain less than 48% sugars (refers to mainly sucrose and invert sugar) and 79.5% dry substance (*DS*) when offered to customers. Such a molasses contains a purity of about 60% or lower, which is a satisfactory molasses in sugar terms. In addition, molasses in the market is sold based on its weight, meaning the sugar factory does not get any credit for sugar content above the limit. Therefore, sugar left in molasses above the limit is considered a **double sugar loss**. Most important, sugar in molasses is valued one-fourth to one-sixth the price of granulated-refined sugar.

Before we get to the details of molasses exhaustion, you might be wondering why molasses has to be formed, so the subject of molasses formation is discussed next.

MOLASSES FORMATION

In the final stage of crystallization (low-raw crystallization), the nonsugar load in the mother liquor (molasses) and its viscosity increase to the extent that further maintenance of **supersaturation** (the driving force of crystallization) is not possible. High **viscosity** (the opposing force of crystallization) creates

- A difficult situation for crystallization
- A long requirement time for crystallization
- A difficult situation for massecuite fluidity and centrifugibility

As stated in previous section (Station 16), sugar crystallization occurs because sucrose molecules relocate themselves from the mother liquor to the crystal structure. In a viscous mother liquid, such as molasses, the movement of sucrose molecules becomes extremely slow, causing a too-low crystallization rate.

High nonsugar content present in the syrup is not the only cause of molasses formation; water is by far the most melassigenic substance (Silin 1958). Let us assume that none of the nonsugars is **melassigenic** and prove that molasses will still form because of water. Because sugar and nonsugars are solids, they are necessarily associated with water to produce a solution. Because sugar and nonsugars increase the dry substance (*DS*) of the solution with their presence, the accumulation of *DS* forms such a viscous solution that further crystallization become impossible. The syrup formed in this way is **molasses**.

Molasses is a solution containing sugar and nonsugars saturated at the final stage of crystallization and 82% *DS*. Assume that in such a solution, nonsugars would have no effect on sucrose solubility and low-raw centrifuging was performed at saturation and at about 50°C. Such solution contains 100 − 82 = 18% water. Because one part of water dissolves 2.59 parts of sugar at 50°C (from Table A.2 in the Appendix), this solution would have $18 \times 2.59 = 46.6\%$ sucrose. Therefore, the purity (*P*) of this molasses would be

$$P = \frac{S}{DS} \times 100 = \frac{46.6}{82.0} \times 100 = 56.8\%$$

With a similar calculation, it is possible to prove that in the presence of any melassigenic nonsugar, molasses will also be formed, but with a higher purity because water is highly melassigenic (*water is the most melassigenic substance and purity-increaser in molasses*).

Because of high melassigenity, water should be removed as much as possible to produce molasses with lower purity. However, water removal is limited by the viscosity (*the lower the water content, the higher is the viscosity*). Viscosity, among other factors, determines the processability of the massecuite and slows the crystallization rate (*the higher the viscosity, the lower is the crystallization rate*). All these factors, referred to as the **melassigenic effect** (molasses-producing actions), affect the process such that further crystallization of such low-purity syrup becomes almost impossible (or, at least requires an unreasonably long crystallization time). The syrup existing with such properties is molasses, which should be separated from the process to prevent nonsugar accumulation and increase the processability of crystallization. After all, *the production of molasses is necessary to operate a sugar factory*.

In sugar technology, the *sucrose-to-nonsucrose* ratio (*S/NS*) is called **melassigenic coefficient** (K_M). The melassigenic coefficient is defined as the kg (pound or tons) of sugar present in molasses per 1 kg (pound or ton) of nonsugars. The K_M can be calculated from molasses purity:

$$K_M = \frac{S}{NS} = \frac{P}{100 - P} \tag{3.17.1}$$

EXAMPLE 1

Given:

Molasses purity (P) 60.0%

Calculate:

K_M

$$K_M = \frac{S}{NS} = \frac{P}{100 - P} = \frac{60}{100 - 60} = 1.5$$

As the example shows, molasses has a high *S/NS* ratio of 1.5 even with a satisfactory purity of 60%. This is the reason for the common expression in sugar processing that *at least 1.5 kg of sugar is lost to molasses for each kg of nonsugars.*

Another important ratio that relates to sucrose solubility (and to molasses formation) is the nonsugar-to-water ratio (*NS/W*) in the massecuite (*as the NS/W value changes, so changes the sucrose solubility in the syrup*). This ratio can indicate whether crystallization has reached the final stage. The functional maximum *NS/W* is about 5. When this value is reached, molasses exhaustibility has reached its maximum limit.

EXAMPLE 2

Given:

Dry substance (*DS*) of low-raw massecuite	95.0%
Purity of low-raw massecuite	74.5%

Calculate:

NS/W in the massecuite

$$S = \frac{DS \times P}{100} = \frac{95.0 \times 74.5}{100} = 70.8\%$$

$$\frac{NS}{W} = \frac{DS - S}{100 - DS} = \frac{95.0 - 70.8}{100 - 95.0} = 4.8$$

According to Silin and Silina (Silin 1958), the following statements, known as Silin-Silina method, can be made with regard to the melassigenic actions of nonsugars:

- Each nonsugar influences sucrose solubility differently, resulting in different levels of *S/NS* and, consequently, different molasses purity.
- Water is by far the most melassigenic substance and purity-increaser in molasses.
- Cations alone cannot be considered as the measure of the melassigenic coefficient because anions also apply considerable influence. Table 3.17.1 gives the relative melassigenic effect of several nonsucrose substances:

Table 3.17.1 Melassigenic Coefficient of Nonsugars

Melassigenic Coefficient (m)									
NaOH	Na_2CO_3	NaCl	KCl	Betaine	K	Na	Ca	Mg	Invert sugar
4.61	2.88	2.58	2.48	1.03	1.00	0.95	0.66	0.61	0.19

Because molasses formation is unavoidable, the sugar factory must *produce molasses with as low a purity as possible to minimize the sugar loss in molasses*. Because the production of low-purity molasses is desired, it is necessary to learn how low the purity value should be for satisfactory molasses. In other words, we want to make sure that the sugar boiler is crystallizing all the sugar that is practically possible from the thick juice entering the sugar-end, while producing a molasses with the lowest possible purity (**satisfactory molasses**).

NORMAL AND SATISFACTORY MOLASSES

Normal molasses (standard molasses) is an absolute exhausted (desugared) molasses, which is saturated with sucrose at 40°C and contains 82% *DS*. The purity of normal molasses changes during a campaign because of changes in the amount and nature of nonsugars in the beets (due to changes in beet quality and the efficiencies of juice purification). Silin (1958) prepared a procedure, called the **Silin test,** that is complicated (requires complicated equipment) and time-consuming (requires one to four days for the molasses to reach saturation). After all, the results are usually questionable, mostly because of the change in the nonsucrose-water ratio (*NS/W*) during the test.

Wagnerowski (Dobrzycky 1973) suggested a "Polish test" for finding the purity of normal molasses. This test is based on the saturation of molasses by dissolving sugar in it at a constant temperature. The test takes less time to run (about eight hours to reach a good equilibrium) and needs a skilled analyst. However, the fact that the normal purity test has to be run on a regular basis and still does not provide the kinetic aspect of the low-raw crystallization convinced many sugar factories not to include either tests in their laboratory schedule. Therefore, it is sufficient to take the purity of 58 as **normal purity** as Silin suggests (Silin 1958).

Satisfactory molasses (practical molasses) is a well-exhausted molasses with the lowest practical purity that can be produced in a given factory with adequate methods and equipment. Typically, satisfactory purity is 59 to 60%, which is slightly higher than normal purity (standard purity) by 1 to 3 units, again as Silin suggests. However, the purity may be reduced below this limit. Satisfactory molasses has approximately 82% dry substance, and its production (usually 4 to 5% OB) is a function of the thick-juice purity.

Because of the property of soda ash as a molasses-purity-increaser (melassigenic), sugar factories utilizing soda ash (soda-ashed sugar factories) cannot produce molasses with as low a purity as the non–soda-ashed factories. However, many factories use soda ash and still produce molasses with satisfactory purity of about 58%. Two such factories are Minn-Dak and Bay City in the United States. Bay City adds 3.5 kg (8 lb) soda ash per ton beet and produces molasses with around 58% purity. (Note: The result as such is achievable if the soda ash dosage is carefully watched because soda ash is highly **melassigenic**.)

The valid question here is why the factory cannot produce molasses with as low a purity as normal molasses. Experience shows that almost all factories deviate from the normal value

by 1 to 3 purity units. One of the reasons for this deviation is that molasses purity is the result of a compromise between the operational conditions in the crystallization and centrifuging process. In a good low-raw (C) crystallization job, the mother liquor (molasses), in the massecuite, leaving the cooling crystallizer, is saturated (has a supersaturation coefficient, K_{SS}, of 1.0), but the massecuite must be heated and sometimes a little diluted to improve its centrifugibility. This cannot be accomplished without a partial remelting of the crystals, which disturbs the K_{SS} and, consequently, increases the molasses purity.

DAMAGE OF HIGH-PURITY MOLASSES

To find out how high-purity molasses can hurt the profitability of a sugar factory, we should know the parameters needed to calculate sugar losses in molasses. Sugar losses in molasses (S_M) can be calculated from the following equation:

$$S_M = (S_C - S_L)\frac{100 - P_{TJ}}{P_{TJ}} \times \frac{P_M}{100 - P_M} \tag{3.17.2}$$

With the processing losses (sugar losses to pulp, carbonation-lime residue, and unaccounted) S_L = 0.6% on beet (OB) inserted as a constant into Eq. (3.17.2), the S_M can be calculated as

$$S_M = (S_C - 0.6)\frac{100 - P_{TJ}}{P_{TJ}} \times \frac{P_M}{100 - P_M} \tag{3.17.3}$$

S_M	Sugar in molasses (% OB)
S_C	Sugar in cossette (%)
P_{TJ}	Purity of thick juice
P_M	Purity of molasses

The example below shows how to calculate the damage to a sugar factory that produces a molasses with an unsatisfactory purity of 63% compared with a satisfactory purity of 60%. Study the example.

EXAMPLE 3

Given:

Factory's capacity	5000 t beets/day
Sugar content of cossettes (S_C)	17.0%
Purity of thick juice (P_{TJ})	90.0%
Purity of molasses (P_M)	63.0%

Calculate:

Sugar loss to 63-purity molasses

Sugar loss to 60-purity molasses

Extra sugar loss due to production of molasses with higher purity

For 63-purity molasses, sugar loss to molasses (% on beet) is

$$S_M = (17.0 - 0.6)\frac{100 - 90.0}{90.0} \times \frac{63.0}{100 - 63.0} = 3.1\%\ \text{OB}$$

$$5000 \times \frac{3.1}{100} = 155\,\text{t/day}$$

For 60-purity molasses, S_M is

$$S_M = (17.0 - 0.6)\frac{100 - 90.0}{90.0} \times \frac{60.0}{100 - 60.0} = 2.7\%\ \text{OB}$$

$$5000 \times \frac{2.7}{100} = 135\,\text{t/day}$$

The extra sugar loss to molasses caused by an increase in molasses purity is equal to the difference in sugar loss between the two cases:

$$155 - 135 = 20\,\text{t/day}$$

If you multiply the tons of extra sugar loss (20 t per day) by the price of sugar, you will be surprised how much money a factory with a capacity of 5000 t/day can lose if they produce molasses with a purity three units higher. (Simply speaking, *production of molasses with high purity is the worst mistake that any sugar factory can make*.)

PRODUCTION OF MOLASSES WITH SATISFACTORY PURITY

As stated, the production of molasses with satisfactory purity is the key to the profitability of any sugar factory. For a given factory and beet quality, the limiting variables of the crystallization operation are the purity of the standard liquor and the **target purity** for satisfactory molasses. Each factory can target a range for purity of molasses based on beet quality and equipment. Let us consider this subject in the context of the following three rules:

- Rule of purity drop
- Rule of backboiling
- Rule of massecuite concentration

Rule 1, Purity Drop

Purity drop (PD, also called the **crystallization effect**) is defined as the purity difference between massecuite (practically, the same as feed to the pan—see the following Notes) and mother liquor centrifuged from the massecuite. For example, if the white massecuite purity is 92% and, after finishing the crystallization and centrifuging, its mother liquor (high-green syrup) has a purity of 86, the PD is 6 units. As you know, in a three-stage boiling, the process continues in the high-raw (B) and low-raw (C) pans until it reaches the mother liquor from the third stage, which is molasses. Therefore, in a three-stage crystallization, we talk about three purity drops, indicated as, for example, 6–10–16, which refers to the PD in the first, second, and third stages of crystallization, respectively.

In general, the purity drop (PD) can be used to determine how efficiently the crystallization process is operating because *the higher the PD, the higher is the crystallization efficiency*. To produce, for example, a satisfactory molasses of 60, an overall PD of 32 is required if boiling starts with standard-liquor purity of 92. The overall PD of 32 is easily achievable by a 6–10–16 PD profile in individual stages of crystallization.

After discharge from the pan, the massecuite is characterized by the following known parameters, obtained from lab data:

- Dry substance of the massecuite (DS_M)
- Purity of the massecuite (P_M)
- Purity of the mother liquor (P_{ML})
- Temperature of the massecuite (T_M)

From the preceding data, the PD is simply determined as $P_M - P_{ML}$. (Note: The **purity** of the massecuite is the same as the purity of the feed (P_F) to the pan because *purity does not change with concentration, dilution, or crystallization.*)

One of the key points in the production of satisfactory molasses is to achieve the right PD at each stage of the crystallization process. Generally, a 6–10–16 to 8–12–18 profile must be targeted. This means 6 to 8 PD units in the first stage of crystallization (white), 10 to 12 units in the second stage (high-raw), and 16 to 18 units in the third stage (low-raw) should be achieved to produce satisfactory molasses of 60% or below from a starting purity of 90 to 94% (refers to standard-liquor purity). Table 3.17.2 shows the maximum allowable low-raw massecuite purity to achieve a selected molasses purity at the given yield.

The massecuite purity at a given *DS* determines:

- Crystal content
- Crystallization efficiency
- Crystallization rate (crystal growth)

The massecuite crystal content (*CC*) is important in an efficient crystallization process. In actual factory operations, it is usually possible to achieve a *CC* of about 35% in the low-raw massecuite (at the discharge from the pan and entering the cooling crystallizer), of about 55% *CC* in white pan, 45 in high-raw pan, and of about 35% in low-raw pan. (Note: The low-raw massecuite gains about 5% in *CC* during cooling-crystallization process, reaching 40%.)

If we know the target for *CC* of the massecuite, its purity (P_M), and its mother-liquor purity (P_{ML}), we can calculate the dry substance (concentration) of the massecuite (DS_M):

$$DS_M = \frac{CC\,(100 - P_{ML})}{P_M - P_{ML}} \tag{3.17.4}$$

Table 3.17.2 Permissible Purity of Low-raw Massecuite

Molasses Purity (%)	Permissible crystal content (%)				
	38	40	42	44	
54	73.2	74.2	75.2	76.1	Massecuite purity
56	74.4	75.4	76.3	77.2	
58	75.5	76.5	77.3	78.2	
60	76.7	77.6	78.4	79.3	
62	77.9	78.7	79.5	80.3	
64	78.9	79.7	80.6	81.4	
66	80.1	80.8	81.6	82.4	

EXAMPLE 4

Given:

High-raw massecuite crystal content target	48.0%
High-raw massecuite purity	87.0%
High-green syrup purity	73.0%

Calculate:
Massecuite dry substance (DS_M)

$$DS_M = \frac{CC\,(100 - P_{ML})}{P_M - P_{ML}} = \frac{48.0\,(100 - 73.0)}{87.0 - 73.0} = 92.6$$

In Figure 3.17.2, Eq. (3.17.4) is plotted for different high-raw massecuite purities with a target crystal content (*CC*) of 48%. To achieve the target *CC* in a 87 purity massecuite, a massecuite *DS* of 92.6% is required. If the massecuite purity is lowered to 85, the massecuite *DS* must be 93.5 to obtain the same crystal content.

In low-raw (*C*) massecuite, the expected target crystal content in the massecuite *at the discharge* from the pan is lower than the high-raw massecuite, because of the lower purity of the massecuite. Figure 3.17.3 shows the required dry substance at a given low-raw massecuite purity to achieve 33% crystal content.

With respect to the relation between *DS* and the purity of the massecuite, the same rule applies to boiling low-raw massecuite. As seen in Figure 3.17.3, to achieve the target *CC* of 33% (at the discharge from the pan) in a 75 purity low-raw massecuite, a massecuite *DS* of 94.0 is required. If the massecuite purity is lowered to 74, the massecuite *DS* must be 94.5 to achieve the same crystal content.

Generally, *lower purity massecuite requires a higher dry substance to obtain the same crystal content.* This is the reason why the dry-substance target of the low-raw massecuite must always be higher than that of the high-raw massecuite. Given the limitation of maximum crystal content, with respect to massecuite purity and fluidity, each factory must establish an ideal purity-drop profile to achieve the desired overall crystallization efficiency and satisfactory molasses purity.

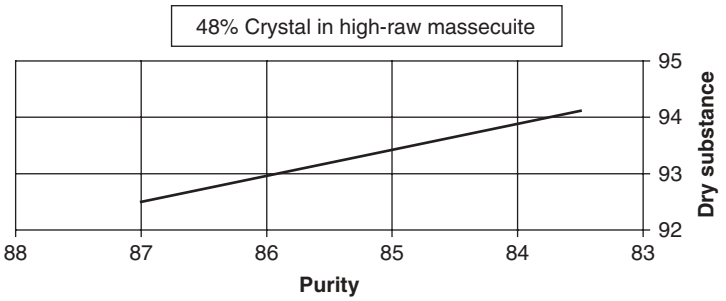


Figure 3.17.2 Effect of high-raw massecuite purity on crystal content

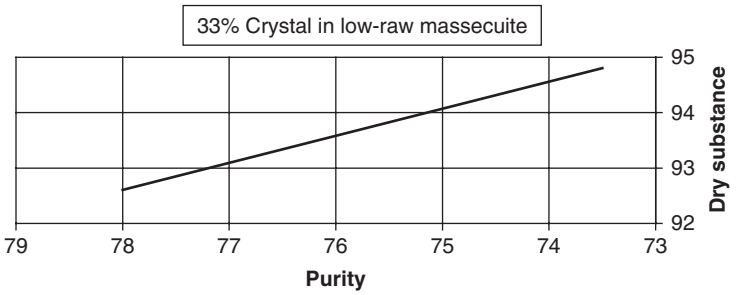


Figure 3.17.3 Effect of low-raw massecuite purity on crystal content

NOTES

- **When we talk about the purity of the massecuite, it should be considered the same purity as the feed to the pan because purity does not change by concentration, dilution, or crystallization (in the case of crystallization, the purity refers to the purity of the massecuite), assuming that no decomposition of sucrose occurs.**
- **The crystal content (*CC*) of the low-raw massecuite after leaving the cooling crystallizer is higher by about 7 to 8% than when discharged from the pan, making the optimum *CC* about 40%. The low-raw massecuite with a *CC* of above 40% must be avoided because it contributes to fluidity problems.**

Rule 2, Backboiling

Backboiling (boiling back or reboiling), in the sugar boiler's language, means mixing lower and higher purity syrup to produce a desired purity feed for a given boiling (crystallization) stage. Backboiling covers the disadvantage of *straight three-stage crystallization* (or four-stage crystallization) when standard liquor has a high purity. It allows the sugar boiler to control the purity of the massecuite by adjusting the purity of feed. For example, when the purity of high-green (A) syrup is high, the sugar boiler adds a certain amount of low-green (B) syrup to it for feed to the high-raw pan or can start feeding the pan with high-green syrup and charging with low-green syrup. This is called backboiling of low-green syrup into the high-raw pan.

In backboiling, the lowest purity syrup should be fed to the pan near the end of the strike to promote the best crystal growth rate during the beginning of the crystallization process. This improves the overall crystallization rate (R_C) and the quality of crystals.

In a crystallization process with a well-used backboiling, the purity of the standard liquor, as the starting purity, cannot make that much difference in producing satisfactory molasses, unless the purity of the standard liquor is too high (above 93%) or too low (below 88%). When you process standard liquor with normal purity (90 to 92%), you can generally control the purity of the raw-side massecuites (low-raw and high-raw) *independently* of the standard liquor purity. In this case, the starting target-point becomes the purity of high-raw massecuite, which

must be closely controlled to produce satisfactory molasses. The high-raw massecuite purity, as the starting target-point, should be in the range of 84 to 86%. With this strategy, any change in the standard-liquor purity is handled in the high-raw pan by adjusting its feed (high-green syrup) purity by backboiling.

When the standard-liquor purity is high, the backboiling consists of blending high-green with low-green syrup for feed to the high-raw pan to produce massecuite with 83 to 86% purity. (Note: This is possible only if sufficient low-green syrup is available.) When the standard-liquor purity is very low, such as in the case of damaged beets, backboiling involves bypassing standard liquor (or thick juice) around white boiling to the high-raw pan. In either case, the purity of the high-raw (B) massecuite should not vary significantly from the target point of 83 to 86%, because *the lower the purity of the starting target-point, the lower is the purity of the mother liquor throughout the crystallization stages and, consequently, the lower is the molasses purity.*

Figure 3.17.4 compares the results of an operation in which backboiling is being used to control the purity of the high-raw massecuite to a nonbackboiling operation. In Figure 3.17.4, the high-raw massecuite purity is lowered from 87 to 85% by backboiling. Because of backboiling, the purities of the low-raw massecuite and molasses are lowered also by approximately the same number of purity units as indicated for the high-raw massecuite. Under normal operating conditions, control of the high-raw massecuite purity at 83 to 86% will result in a low-raw massecuite of about 72 to 75% purity and a satisfactory molasses of 58 to 60% purity. Therefore, one can state that because of backboiling, the *control of molasses purity is largely achievable through the control of the high-raw (B) massecuite purity.*

In backboiling, the required volume of mixing syrups (V_1 and V_2) can be determined by the dry substance (DS_1 and DS_2) and purity (P_1 and P_2) of the syrups to achieve a required volume of massecuite (V_M) with the desired dry substance (DS_M) and purity (P_M) by the following equations:

$$V_1 = \frac{DS_M(P_M - P_2)V_M}{DS_1(P_1 - P_2)} \tag{3.17.5}$$

$$V_2 = \frac{DS_M(P_M - P_1)V_M}{DS_2(P_2 - P_1)} \tag{3.17.6}$$

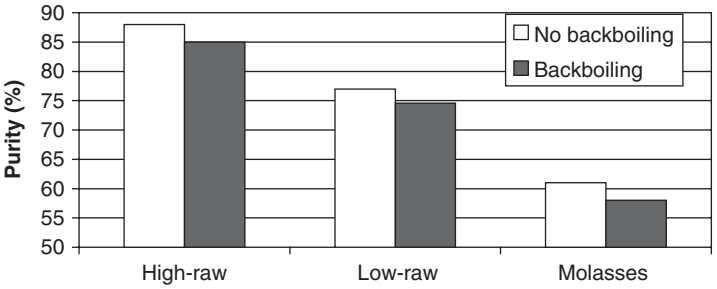


Figure 3.17.4 Raw-side purity profile with and without backboiling

EXAMPLE 5

Given:

Amount of high-raw massecuite required (V_M)	50 m³ (1770 ft³)
Dry substance of high-raw massecuite (DS_M)	93.0%
Dry substance of high-green syrup (DS_1)	76.0%
Dry substance of low-green syrup (DS_2)	80.0%
Purity of desired massecuite (P_M)	86.0%
Purity of high-green syrup (P_1)	88.0%
Purity of low-green syrup (P_2)	77.0

Calculate:

Volume of high-green syrup (V_1) and low-green syrup (V_2) and their percentage to be mixed to produce high-raw massecuite with the desired purity of 86.0%

$$V_1 = \frac{DS_M(P_M - P_2)m_M}{DS_1(P_1 - P_2)} = \frac{93.0(86.0 - 77.0)50}{76.0(88.0 - 77.0)} = 50.1\,\text{m}^3(1770\,\text{ft}^3)$$

$$V_2 = \frac{DS_M(P_M - P_1)m_M}{DS_2(P_2 - P_1)} = \frac{93.0(86.0 - 88.0)50}{80.0(77.0 - 88.0)} = \frac{-9300}{-880} = 10.6\,\text{m}^3(374\,\text{ft}^3)$$

$$V_1 + V_2 = 50.1 + 10.6 = 60.7\,\text{m}^3\ (2144\,\text{ft}^3)$$

$$V_1 = \frac{50.1}{60.7} \times 100 = 82.5\%$$

$$V_2 = 100 - 82.5 = 17.5\%$$

NOTE

The sugar boiler does not need to go through these calculations every day. Given steady operating conditions and thick-juice purity and using a good rule of thumb, the sugar boiler can estimate to backboil such percentage of low-green syrup into the high-raw pan to produce high-raw massecuite to meet the starting target-purity (83 to 86%) Example 4 in the Appendix makes the calculation of backboiling easier.

Rule 3, Massecuite Concentration

Because of high melassigenity, water should be removed as much as possible to produce molasses with lower purity. However, water removal from the syrup under crystallization is limited by the viscosity (*the higher the viscosity, the lower is the crystallization rate*).

The concentration (dry substance, *DS*) of the massecuite leaving the pan has a direct effect on the crystal content (yield) from the massecuite. If *DS* is high, the crystal content (*CC*) will be high, and if *DS* is low, the *CC* will be low too. On the other hand, when massecuite purity is low, considerable time is required to produce sufficient crystal content and suitable concentration of the massecuite. Why is this true? The answer lies in **sucrose solubility** in water (as well as in impure solutions).

As seen in Figure 3.17.5, at a typical temperature of 75°C, sucrose solubility is 3.5, meaning 1 kg water contains 3.5 kg of sugar. Therefore, *for 1 kg of water evaporated from the massecuite, about 3.5 kg of sugar is transferred from the syrup to the crystals*. The evaporation of water from the massecuite results in a higher *DS* (less water). The fact that less water is present in the massecuite means that more sugar was transferred from the liquid to solid (crystal) phase. This means that the purity of the mother liquor in the pan is lowered, resulting in higher sugar yield (lower mother-liquor purity) from the crystallization operation. In sum, the production of massecuite with higher *DS* (to a practical level) in individual stages of crystallization results in a lower-purity mother liquor (respectively, molasses purity). Such an achievement has a large effect on the efficiency of the factory.

As Figure 3.17.6 shows, the *DS* of the high-raw massecuite has been increased from 92.0 to 92.8. As a result, the mother-liquor purity has dropped by 1.5 units and the crystal content has increased by 6%, a considerable amount.

The fact that less water is present in the massecuite means that more sugar was crystallized (more crystal content). The sugar boiler wants to produce the massecuite with the highest possible crystal content (*CC*) but knows that there is a limit to it because a massecuite with high *CC* (actually with high *DS*) does not flow well enough to be moved through the process. The practical limit of *CC* is about 55% at the first stage of crystallization, 50% at the second stage, and 33% at the third stage.

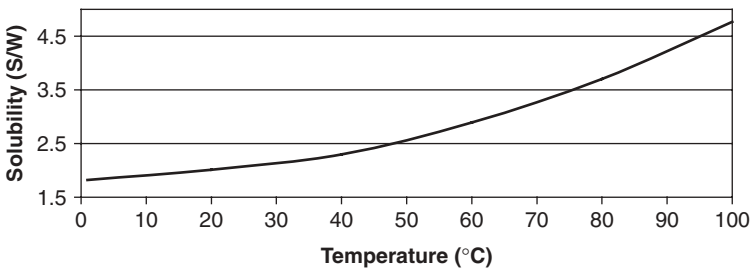


Figure 3.17.5 Sucrose solubility at different temperatures

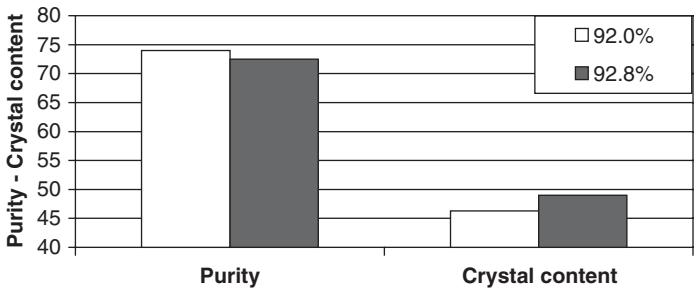


Figure 3.17.6 Effect of massecuite *DS* on mother-liquor purity and massecuite crystal content

The relation between massecuite viscosity and its concentration will be discussed later. However, it should be obvious now that as the massecuite dry substance is increased, the crystal content increases and the mother-liquor purity (respectively, molasses purity) decreases proportionately. In other words, *the higher the massecuite concentration (dry substance), the lower the mother-liquor purity and consequently the lower the molasses purity.*

A change in the purity or the *DS* can change the *NS/W* value. In sugar-end operations, usually a change in *DS* changes the nonsugar-to-water ratio ***NS/W***. As *DS* increases, the amount of water in the molasses is reduced (*water dissolves more sugar than nonsugars do*), so the *NS/W* value increases. Therefore, the potential amount of sugar in molasses is lower, given the same temperature and supersaturation. Having said that, high *NS/W* decreases sucrose loses in molasses (decreases molasses purity) mostly because of the reduction in water content of the molasses.

Generally, sugar-end operations can be targeted according to the values given in Table 3.17.3.

Effect of Viscosity on Low-Raw Crystallization

In the previous section (Station 16), **viscosity** (a measure of the resistance of a liquid to flow) and its general effect on the crystallization process (rate) was discussed. Now, you need to learn the role of viscosity as a **limiting factor** in low-raw crystallization.

At the same *DS*, as massecuite gets to further stages of crystallization, its mother liquor gets more viscous. Molasses, as the mother liquor of the final stage of crystallization, is the most viscous liquid in the sugar operation (it flows at a very slow rate). The higher dry substance (at saturation) causes the higher viscosity. The following generalizations are true for relatively low-purity (high nonsugars, *NS*) syrups:

Increase in temperature = Decrease in viscosity

Increase in dry substance = Increase in viscosity

Increase in purity (decrease in *NS*) = Increase in viscosity

For molasses at about 60°C, 82% *DS*, the following are true:

+1°C temperature = –10% viscosity

+1% dry substance = +50% viscosity

+1% purity (almost –1% *NS*) = +1% viscosity

As stated in Section 16 of this chapter, viscosity depends mainly on the temperature, dry-substance content, purity (nonsucrose content) of the solution, and to a minor extent on the pressure (except at very high pressure) of the solution. Figure 3.17.7 shows the relationship between viscosity (in centi poise, cp) and dry substance in molasses with 60% purity at 50°C.

Table 3.17.3 A typical Sugar-End Target Profile

	Dry-Substance (%)	Purity (%)	Purity Drop	Crystal Content (%)
White massecuite	93.0	92.0		55.0
High-raw massecuite	94.0	84.0	8.0	50.0
Low-raw massecuite	95.0	74.0	10.0	33.0
Molasses	81.0	58.0	16.0	

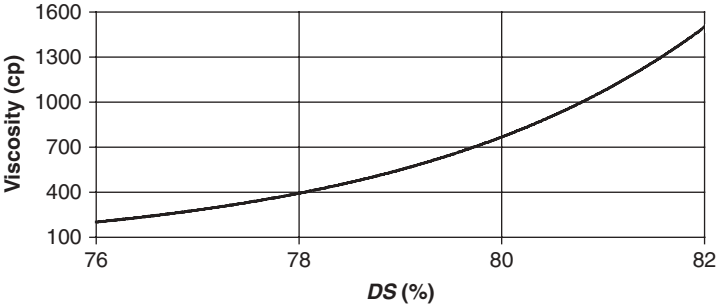


Figure 3.17.7 Effect of dry substance on viscosity of 60-purity molasses at 50°C

Because the *DS* of the massecuite is a direct function of its *crystal content* and *crystal size*, effects of these factors on viscosity should be well understood as it is discussed next.

Effect of Crystal Content and Crystal Size on Viscosity

The size and content of crystals in a massecuite affect its flowability. As the concentration of crystals increases in a massecuite, its viscosity (resistance to flow) increases. In general, it is the viscosity of the massecuite that creates difficulties in the crystallization process, not the viscosity of the mother liquor. (By itself, molasses will usually flow at any reasonable process temperature, but if we add crystals to molasses, the flow becomes much slower.)

Figure 3.17.8 shows the influence of crystal content (*CC*) on the viscosity of low-raw massecuite. A fast increase in the viscosity occurs when *CC* is more than 40%. The scale on the left axis of the graph gives the ratio of the massecuite viscosity to the syrup viscosity.

NOTES

- **For crystal yield and sugar recovery, the stiffer the massecuite the better. But for flowability, there is a limit to the maximum crystal content in the process. This limit is about 52% crystals in white massecuite, about 48% in high-raw massecuite, and about 36% in low-raw massecuite (exiting the cooling crystallizer). The reductions in values are due to the higher viscosity of the syrup in the low-purity massecuites.**
- **When the low-raw massecuite purity is very high (above 77%), the crystal content in the massecuite may be increased to about 42%.**
- **Strikes intended for seed magma should generally be boiled at a lower *CC* (lower final dry substance). The proper final *DS* of the magma (massecuite) for seeding depends on the type of boiling scheme employed (typically 91% *DS*).**

Also important for viscosity is the **crystal size** in the massecuite. For a given weight of crystals in a massecuite, larger crystals yield a massecuite with lower viscosity. This fact has significant importance in the optimization of sucrose recovery. (Note: A larger crystal size results in significantly lower massecuite viscosity for a given yield.) This phenomenon strongly suggests that *larger crystals increase crystal yields for any step in the crystallization process*. For this reason, many

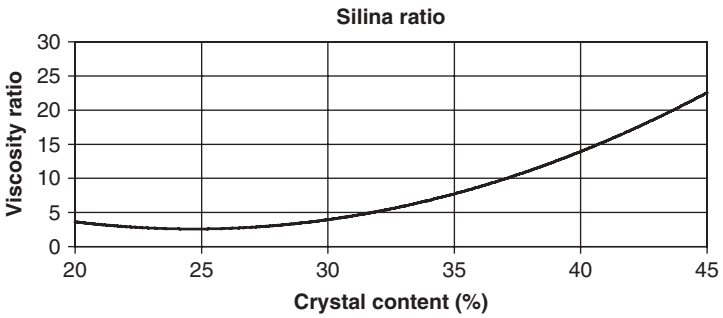


Figure 3.17.8 Effect of crystal content on viscosity of low-raw massecuite

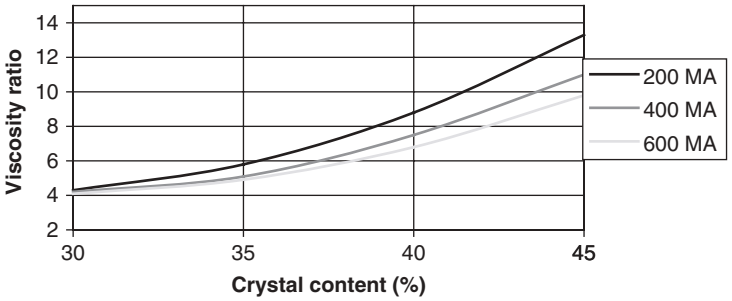


Figure 3.17.9 Effect of crystal size on viscosity

sugar factories produce high-raw massecuite and low-raw massecuites with large crystals to optimize the capacity, recovery, and fuel consumption of the sugar-end. This fact is particularly important because there are *no limitations on crystal size of raw massecuites with respect to customer specifications*.

Figure 3.17.9 shows the influence of crystal sizes of 0.2, 0.4, and 0.6 mm (200, 400, and 600 microns or *MA*, where *MA* stands for **mean aperture**) on the viscosity ratio (massecuite viscosity to mother liquor viscosity). In addition to the positive effect that larger crystals have on massecuite fluidity, it is important to note that for any given crystal content, *larger crystals* mean a *smaller crystal surface area*. (Note: The smaller surface area, while having a slight negative effect on the overall crystallization rate, largely reduces centrifugal washing requirements.)

HIGH-RAW AND LOW-RAW CRYSTALLIZATION

The general procedure for high-raw and low-raw crystallization is almost the same as that for white-massecuite crystallization, which was explained in detail in Station 16. In raw-side crystallization, particularly in low-raw, you should consider few points to produce quality raw sugar with the right uniform size and crystal content (*CC*):

- High-raw massecuite is boiled to a *DS* of about 95% and low-raw massecuite to about 96%.

- The high-raw pan is boiled first in the range of 25 to 32% *CC* and concentrated to a final *CC* of about 48%. The low-raw pan is boiled in the 20 to 28% range and concentrated to 32 to 34% crystal at drop. Cooling crystallization will then increase the *CC* to about 40%.
- The boiling time of second crystallization is 4 to 8 hours, and the third crystallization takes 8 to 12 hours, mainly because of the difference in syrup purity (*the lower the purity, the lower is the crystallization rate*). Seed size, supersaturation coefficient (K_{SS}), and pan circulation are other factors that affect the crystallization rate (boiling time).

In general, syrup with low purity has the following undesirable effects:

- Decreases the crystallization rate
- Decreases the mobility of the molecules
- Increases the viscosity of the massecuite

After discharge from the pan, the low-raw massecuite is cooled in the cooling crystallizer to increase its *CC* (from about 33 to 40%) and decrease molasses purity. Under good operation conditions, molasses reaches its lowest practical purity after cooling crystallization. At this point, it must be separated from the process to prevent nonsugar (impurity) accumulation.

As explained in Section 16, some modern sugar factories use **continuous crystallizers** in high-raw and low-raw crystallization. When operating continuous crystallizers, particularly the **VKT** (BMA continuous vertical crystallizer), for *high-raw boiling*, you should control the following:

- **Seed preparation: Seed magma** is produced by mixing the low-raw sugar with green syrup (mostly low-green syrup). The purity target range for seed magma for high-raw boiling is approximately 84%. This matches the purity target of the high-raw massecuite. You can produce seed magma also by remelting low-raw sugar directly into thick juice. In the case of insufficient low-raw sugar, additional batch seed strikes can be boiled to maintain a sufficient supply of feed. On the other hand, excess seed magma to the continuous crystallizer (pan) reduces its capacity because of reduction of the retention time in the crystallizer for normal crystal growth. Seed magma should have the same purity as the high-raw massecuite and approximately 91% *DS*. This type of seed usually will produce a high-raw massecuite with a crystal size of about 0.5 mm (500 microns or 500 *MA*).
- **Seed-to-feed ratio:** The seed-to-feed ratio should be at least 25%. If this ratio falls below the minimum amount, insufficient crystals will be in the pan, which may cause false-grain formation.
- **Purity of the massecuite:** As stated earlier, the feed to high-raw boiling is high-green (A-green) syrup. The purity target range for high-raw massecuite is approximately 85%. If the purity is above the target range, backboiling is helpful in controlling the massecuite purity. The low-green (B-green) syrup can be mixed with the high-green syrup entering the last two chambers of the continuous pan.
- **Speed of the process:** The speed of the process is controlled by the amount (or pressure) of the steam supplied to the chamber steam chest (calandria). If you increase the steam pressure, the pan operation speeds up.
- **Dry substance of the massecuite:** The *DS* profile of the VKT for high-raw boiling in individual chambers should be controlled similarly to batch high-raw crystallization at different stages. First chamber's *DS* is similar to a batch boiling at 45 to 60 minutes after seeding and

chamber two at 90 to 120 minutes. Chamber three *DS* is similar to batch boiling when the final concentrating begins, and chamber four is like when the strike ends. Table 3.17.4 shows a typical *DS* profile of low-raw VKT in individual chambers.

When operating low-raw boiling, you should control the following:

- **Seed preparation:** Seed massecuite is produced by batch boiling, using some combination of high-green (A-green) and low-green (B-green) syrup to achieve the desired seed magma and final low-raw massecuite purity. All the high-green syrup should be used in the beginning of seed boiling because it promotes better crystal formation and growth. You should manage the amount of high-green and low-green syrup to produce seed massecuite with about 75% purity. Seed massecuite should have approximately 90 to 91% *DS*. The purity target for seed massecuite is approximately 75%, which matches the purity target of the low-raw massecuite at the discharge. Table 3.17.5 gives an example of the purity of seed massecuite required to achieve a 75% purity low-raw massecuite at various low-green purities. Ideally, seed massecuite should have a crystal size of 0.2 mm (200 microns or 200 *MA*). This will produce a low-raw massecuite with a crystal size of about 0.3 mm (300 *MA*).
- **Seed-to-feed ratio:** The seed-to-feed ratio should be about 35%.
- **Purity of the massecuite:** In general, you a*djust massecuite purity by adjusting seed-massecuite purity*. The purity target for low-raw massecuite is approximately 75%.
- **Speed of the process:** The same points described for the high-raw pan applied here.
- **Dry substance of the massecuite:** Table 3.17.6 shows a typical *DS* profile of low-raw VKT in individual chambers.

AFFINATION

Affination is the process of mixing raw sugar or low-purity massecuite with high-purity syrup and then centrifuging it to improve sugar quality. Mostly, low-raw sugar is affinated using low-green or high-green syrup. Many factories use affination, particularly when their raw-side faces high-color syrups. Following are the benefits of affination:

Table 3.17.4 Typical *DS* Contents in Individual Chambers of High-Raw VKT

	Seed	Ch. 1	Ch. 2	Ch. 3	Ch. 4
Massecuite *DS*	90.0	90.0	90.5	91.5	92.5

Table 3.17.5 Low-Raw Seed Purity Guidelines

	Purity (%)					
Low-green syrup	71.0	72.0	73.0	74.0	75.0	76.0
Seed massecuite	82.8	80.8	76.9	76.9	75.0	73.1

Table 3.17.6 Typical *DS* Contents in Individual Chambers of Low-Raw VKT

	Seed	Ch. 1	Ch. 2	Ch. 3	Ch. 4
Massecuite *DS*	91.0	90.3	91.0	93.0	95.0

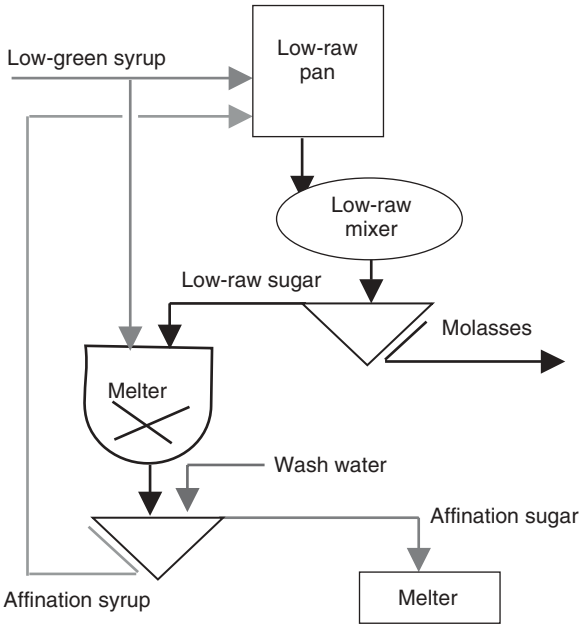


Figure 3.17.10 Low-raw affination

- Increases the purity of mother liquor
- Increases the purity of low-raw sugar
- Decreases the color of low-raw sugar

Because of affination, the purity of the standard liquor is increased and its color decreased. In the affination process (see Figure 3.17.10), the low-raw sugar is mixed with low-green (B-green) syrup to produce a higher purity massecuite. Massecuite is then centrifuged to produce a better quality low-raw (C) sugar. The **affination syrup** (the syrup formed during centrifuging of the affination massecuite) is then sent to the low-raw (C) pan, and the affination sugar is sent to the melter. (Note: When the purity of low-raw sugar is too low, high-green syrup is used for affination.) Refer to Chapter 9 for more information about affination of raw-cane sugar.

With affination, the quality of low-raw sugar is improved at the expense of the nonsucrose recycle to the raw side (high-raw and low-raw), increasing molasses purity (the disadvantage of affination). However, you can achieve the benefits of the affination process and keep the purity of molasses at a reasonable level if you monitor the process closely.

MOLASSES RECYCLE

To maximize crystallization in the pan, sometimes massecuite is boiled to a high *DS* (above 94%). This means the massecuite *NS/W* may be too high (and therefore, the flow too low) for good cooling crystallization performance. To ease this condition, **molasses is recycled** (or water is added) to the massecuite during the cooling process to lower the massecuite *DS* to slightly below 94%. In effect, the recycled molasses lowers the purity and *DS* of the massecuite, improving its fluidity. The advantage of mixing with recycled molasses versus water is that the

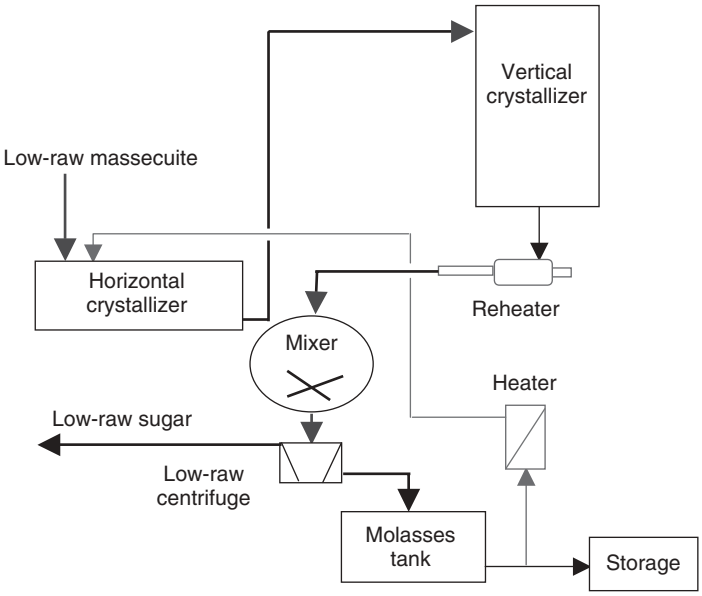


Figure 3.17.11 Molasses recycle system

addition of molasses does not reduce the amount of sugar that can be crystallized from the massecuite. With molasses recycling, you can attain a lower molasses purity than would be possible adding water.

Molasses recycling schemes (see Figure 3.17.11) generally consist of a heating and flow control system that provides a recycle flow of molasses to the feed going to the low-raw cooling crystallizer. The recycle flow is generally controlled by the flow of the feed to the cooling crystallizer to maintain a constant ratio of massecuite to recycled molasses. The greatest disadvantage of the molasses-recycling system is that the molasses must be removed twice in the low-raw centrifuge. If not managed properly, the molasses recycle can quickly consume a large portion of the low-raw centrifugal capacity and limit the processing capacity of the sugar-end.

In the newer approach (shown in Figure 3.17.11), heated molasses is used to condition cold low-raw massecuite before centrifuging. The molasses is heated to about 90°C and fed to a blending device that mixes the hot molasses with the cold massecuite to the desired centrifuging temperature. This type of system provides satisfactory massecuite conditioning and reheating.

COOLING CRYSTALLIZATION

Cooling crystallization is used to finalize molasses exhaustibility by removing all sugar possible from the last mother liquor (molasses). In evaporating crystallization, the released water causes sugar crystals to grow because as the syrup evaporates, less water remains in solution (*solubility decreases with decreasing water content of the massecuite*). In cooling crystallization, the decrease in temperature of the massecuite causes the water in the mother liquor to hold less

sugar (*solubility decreases with decreasing temperature*). As a result, more sugar molecules in the mother liquor position themselves on the crystals as the supersaturation coefficient (K_{SS}) increases with the cooling of massecuite. Because of the cooling crystallization, the crystal content (*CC*) of the massecuite increases, and its mother-liquor (molasses) purity decreases. As stated previously, the low-raw massecuite entering the cooling crystallizer contains about 33% *CC* and improves to about 40% at the discharge. This means lower sugar losses to molasses.

In the final stage of vacuum crystallization in the pan, crystallization practically ceases to occur because of the following conditions:

- High nonsucrose content of the mother liquor
- High *DS* content of the mother liquor
- High viscosity of the mother liquor

When these conditions occur, maintaining supersaturation (refers to K_{SS}) and crystallization become difficult, although the mother liquor still contains a large amount of sugar and crystallization is not completely finished. So the sugar boiler stops the process and discharges the massecuite even though the mother liquor is not completely exhausted.

To maximize mother-liquor exhaustibility, the low-raw massecuite is treated in the cooling crystallizer under atmospheric pressure. When the massecuite is cooled in the cooling crystallizer, excess sugar crystallizes as the temperature drops (*sucrose solubility decreases as temperature decreases*). At 60°C, the sucrose solubility in a 65-purity molasses is 3.3 (from Table A.3, given in the Appendix). As we cool this massecuite in the cooling crystallizer to about 45°C (the usual temperature of the cooling crystallizer), its solubility decreases to 2.7 g per gram of water. The excess sucrose (3.3 − 2.7 = 0.6 g) of the liquor then crystallizes, lowering its purity and, consequently, molasses with lower purity is produced after the massecuite is centrifuged.

During cooling, the dry-substance (*DS*) content of the massecuite remains almost the same (small changes are the result of water evaporation). But the *DS* of the mother liquor decreases during the process because of the relocation of sucrose from the liquor to the crystal phase.

Sugar technologists have studied extensively the relation of the final cooling temperature in the cooling crystallizer with the production of satisfactory molasses. The results of their investigations have led to different recommendations. Compared with the usual cooling-crystallizer temperature of 45°C, further cooling to 35°C and even lower is suggested. Some technologists suggest operating the low-raw massecuite with higher concentration and treating it in the cooling crystallizer with higher temperature. However, most technologists believe that *cooling the massecuite below 45°C rarely results in lower molasses purity* unless a long retention time in the crystallizer is available. This is due to the low crystallization rates because of higher viscosity at lower temperatures. In practice, optimum results can be achieved if the low-raw massecuite is treated in the cooling crystallizer at 45 to 55°C with sufficient retention time.

High retention time (more than 24 hours) in the cooling crystallizer is not economically justified. However, there must be sufficient time and an effective cooling process to crystallize sucrose as much as possible. For a good job in the cooling crystallizer, you should:

- Keep supersaturation sufficiently low to prevent the formation of *false crystals*
- Keep the massecuite under complete and slow stirring
- Keep the massecuite under gradual and fast cooling

In modern crystallizers, most of the cooling is accomplished in the first half of the retention time available. In the final half, the massecuite is held at the low point temperature to maximize molasses exhaustibility and, consequently, sugar crystallinity. Most of the crystallization takes place during the first half of retention time, with the mother-liquor supersaturation coefficient (K_{SS}) approaching 1.4+. The crystallization rate decreases near the end of the cycle where the K_{SS} approaches 1.1 as the massecuite exits the cooling crystallizer.

EXAMPLE 6

Given:

Mother liquor of low-raw massecuite at the beginning of the cooling crystallizer has the following properties:

Temperature (T)	65°C
Dry substance (DS)	84.0%
Purity (P)	64.0%

The mother liquor (molasses) leaving the cooling crystallizer has the following properties:

T	45°C
DS	82.0%
P	60.0%

Calculate:

Supersaturation coefficient (K_{SS}) of molasses entering and leaving the crystallizer

Sugar content (S) can be calculated from the purity equation:

$$S = \frac{84.0 \times 64.0}{100} = 53.8$$

Sugar (S) to water (W) ratio is

$$\frac{S}{W} = \frac{S}{100 - DS} = \frac{53.8}{100 - 84.0} = 5.24$$

According to Table A.3 (in the Appendix), the sucrose solubility at saturation and 65°C in syrup with purity of 64 is 3.64:

$$K_{SS} = \frac{(S/W)_{Act}}{(S/W)_{Sat}} = \frac{5.24}{3.64} = 1.4$$

At the time of leaving the crystallizer, the sugar content of the molasses is

$$S = \frac{82.0 \times 60.0}{100} = 49.2$$

$$\frac{S}{W} = \frac{S}{100 - DS} = \frac{49.2}{100 - 82.0} = 2.73$$

Sucrose solubility at saturation and 45°C in syrup with purity of 60 is 2.87. The K_{SS} at the time of leaving the cooling crystallizer is

$$K_{SS} = \frac{(S/W)_{Act}}{(S/W)_{Sat}} = \frac{2.73}{2.87} = 0.95$$

NOTES

- **It is important to consider the low point temperature for the massecuite exiting the cooling crystallizer. The optimum temperature is in the range of 45 to 50°C, depending on the viscosity of the molasses. Overcooling of the massecuite below this temperature will generally not result in a lower molasses purity because crystallization is severely inhibited by the higher viscosity in overcooled massecuite. In modern crystallizers, a slight increase in the final cooling stage may ever results in better crystallization.**
- **Fast cooling crystallization is the newest method for maximum molasses exhaustibility. Several cooling crystallizers offer this technique. Figure 3.17.12 compares the cooling rates of systems utilizing a slow versus a fast cooling technique. One of the more efficient units of fast cooling crystallization is a design that incorporates two cooling circuits in a single crystallizer (shown in Figure 3.17.12).**

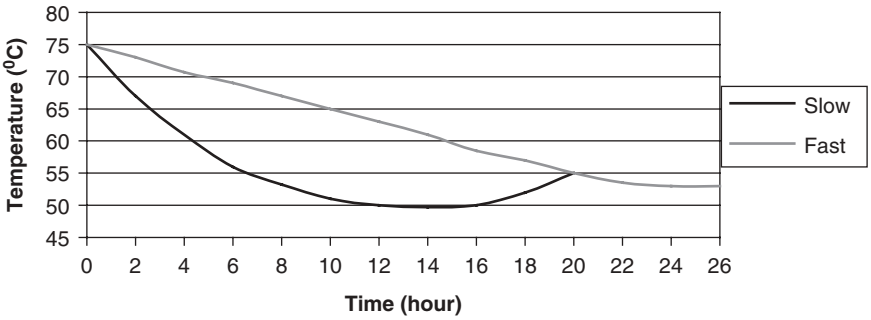


Figure 3.17.12 Comparing slow and fast cooling crystallization techniques

COOLING CRYSTALLIZERS

As in evaporating crystallization, the cooling crystallization is conducted in batches or continuously. In batching of the older type, the boiled massecuite stays in the crystallizer until it gradually cools. Then it is discharged into the distributor and directed to the centrifugal station. The empty crystallizer is refilled with a new batch of massecuite.

Continuous crystallizers are of two types:

- Horizontal
- Vertical

The **horizontal continuous crystallizer** usually consists of a few horizontal tanks connected by chutes. The massecuite enters the first crystallizer and gradually moves to the others, cooling

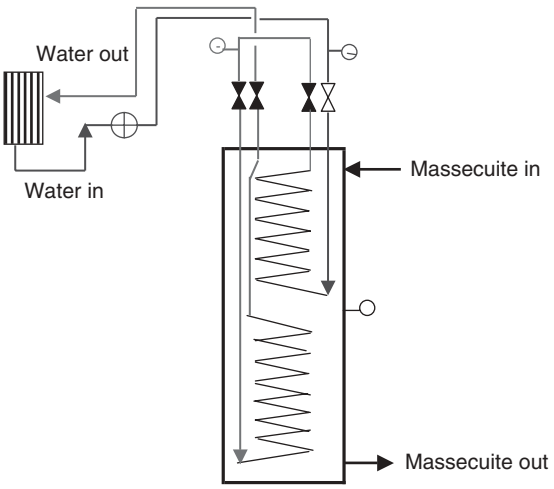


Figure 3.17.13 Cooling design in a vertical cooling crystallizer

on its way until it leaves the last compartment to go to the distributor. Cooling water moves in the cooling element counter to the flow of massecuite. Cold water enters at the coldest end of the crystallizer and leaves the warmer end.

Some horizontal crystallizers cannot handle viscous (stiff) massecuite because of limitation in their drive mechanism. So the *DS* of the massecuite dropped from the vacuum pan crystallizer cannot be as high as required for the optimum crystallization performance of the pan.

The **vertical cooling crystallizers** (see Figure 3.17.13) require less floor space and most are installed outside the factory building. The cooling cycle in a vertical crystallizer is different from a horizontal one. The vertical crystallizer cools the massecuite faster and holds it at a lower temperature for a longer period earlier in the cycle. Typically, vertical crystallizers contain two cooling elements. One is installed in the top half of the unit and the other is in the bottom half. Cooling elements get the cooling water in series, with the top element receiving the coldest water.

MASSECUITE REHEATING BEFORE CENTRIFUGING

Because the **low-raw** (C) massecuite is cooled in the cooling crystallizer (to about 45°C), viscosity of the massecuite becomes high to the point where it is hard to centrifuge. To reduce its viscosity and improve its processability in the low-raw centrifuges, the massecuite is usually reheated to about 55°C. It is important that the reheating be performed in a way that does not redissolve sugar crystals. Massecuite can be reheated by about 10°C without redissolving crystals; this amount of heating only reduces slightly the mother-liquor supersaturation. In other words, almost no loss of crystals from reheating can occur.

Some factories are equipped with a modern reheating system that heats the massecuite quickly by a temperature-controlled water system. The tube design of the heating element ensures a maximum, smooth heating surface (no zone of local overheating) with a minimum retention time of about 30 minutes.

In other reheating designs, heated molasses (about 90°C) maintains the temperature of cold low-raw massecuite before centrifuging. In this system, the molasses is heated to about 90°C and fed to a blending device that mixes the hot molasses with the cold massecuite to increase the massecuite temperature to about 55°C. The massecuite-reheating system is simple, consisting of a motor-driven inline blender that mixes heated molasses with the cold massecuite exiting the cooling crystallizer. The fluidity of the massecuite improves because of the dilution with molasses and increase in its temperature by a few degrees. This provides necessary fluidity required at the centrifugal station. The amount of molasses mixed with the massecuite is small (2 to 3 liters, or 0.5 to 0.8 ga) of molasses per minute for each 1000 t beet processing.

OPERATING PROBLEMS

Molasses is the mother liquor of the last stage of crystallization. After the molasses is discharged from the centrifuge, it is stored in a tank under neutral or slightly alkaline pH conditions to prevent microbial contamination. Beet molasses is the most important by-product of the beet-sugar factories. Satisfactory molasses has a purity of 59 to 60%, with extreme variations from 57 to 63%. Molasses is used in animal feed, yeast, citric acid, alcohol, and pharmaceuticals. Below are some **problems** that can occur in the low-side crystallization operation.

Excessive Foaming during Crystallization

Syrups in the pan make more **foam** at the start of boiling than toward the end of crystallization. Foam occurs mostly in low-raw than in white and high-raw crystallization. Possible causes for high foam content are the following:

- Syrup contains saponin
- Air leak in the vacuum pan
- High speed of the pan agitator
- Damaged beets were processed
- Low addition of defoaming agent
- Low-purity syrup (high nonsugars)
- Poor control of levels in syrup pumping tanks

Slow Cooling Rate of Vertical Cooling Crystallizer

Possible causes for a slow cooling rate in a vertical cooling crystallizer follow:

- **Temperature of cooling water too low:** This problem can be solved by observing the exit water temperature from the upper coil and not allowing this temperature to fall bellow 53°C. The normal operating range of the exit water of the upper coil is 55 to 58°C (depending on the exit temperature from the pan and conditions of the mother liquor *DS* and purity). The exit water temperature of the lower coil is usually lower than this value.
- **Flow rate of massecuite too high for the capacity of the crystallizer**

Required Temperature of Massecuite Not Met

Low-raw massecuite is normally cooled to 48 to 50°C in the cooling crystallizer. However, the massecuite temperature depends on the available retention time in the cooling crystallizer,

the type of the crystallizer, the reheating equipment, and the type of low-raw centrifuges. The temperature of the cooling water supplied to the cooling crystallizer and the flow rate of the massecuite through the process determine the final massecuite temperature. As the feed rate to the crystallizer changes, the temperature of the cooling water must be adjusted to maintain the same final temperature in the massecuite exiting the cooling crystallizer. However, the difference cannot exceed the maximum allowable cooling rate for the massecuite; otherwise, the chance of false crystal formation increases.

NOTE

- **The job of the molasses-exhaustion station is successfully finished when the purity of the mother liquor of the low-raw massecuite reaches the lowest possible to produce satisfactory molasses with about 8.2 to 8.8 pH (molasses pH depends on the use of soda ash and Magox in the purification station). In addition, the massecuite should have the highest possible centrifugibility in order to produce molasses with the lowest possible purity (satisfactory molasses) after centrifuging. In fact, the job of this station is finished after reheating the low-raw (C) massecuite to about 55°C for better centrifuging. Note that the white and high-raw (B) massecuites do not need reheating before centrifuging. At this point, the massecuites are ready for centrifuging in the centrifugal station, which will be discussed in the next section.**

CHAPTER
3

SECTION 18
MASSECUITE CENTRIFUGING

SUBSECTIONS

- Factors Affecting Centrifuging
- Centrifuging of White Massecuite
- Centrifuging of Raw Massecuites
- Double Centrifuging
- Types of Centrifuges
- Operating Problems

ABOUT THIS SECTION **This section** describes **Station 18** (Massecuite Centrifuging) of sugarbeet processing. Station 18 (see Figure 3.18.1) usually consists of three sets of centrifuges: white (A), high-raw (B), and low-raw (C) centrifuges. Centrifugibility of the massecuites and efficiency of the centrifuges mainly determine processability of the massecuite during centrifuging. Among other factors, the crystallization station should produce massecuites with good centrfugibility because the massecuites are then sent to centrifugal station to separate crystals from the mother liquor.

In the last two sections, you learned about crystallization and how it should be performed to produce high-yield sugar and low-purity molasses. You learned that to achieve these goals, the concentration of the massecuite and its crystal content in the pan crystallizer should be increased to the highest practical level. Centrifuging of such viscous massecuite is not possible unless the factory is equipped with efficient centrifuges that create sufficient **centrifugal force** (F_C). (A simple example of centrifugal force is the removal of water from clothing in the spin cycle of a household washer.)

History: Antonin Prandl invented the first centrifuge in early 1700ies for separating cream from milk.

Beet-Sugar Handbook, by Mosen Asadi
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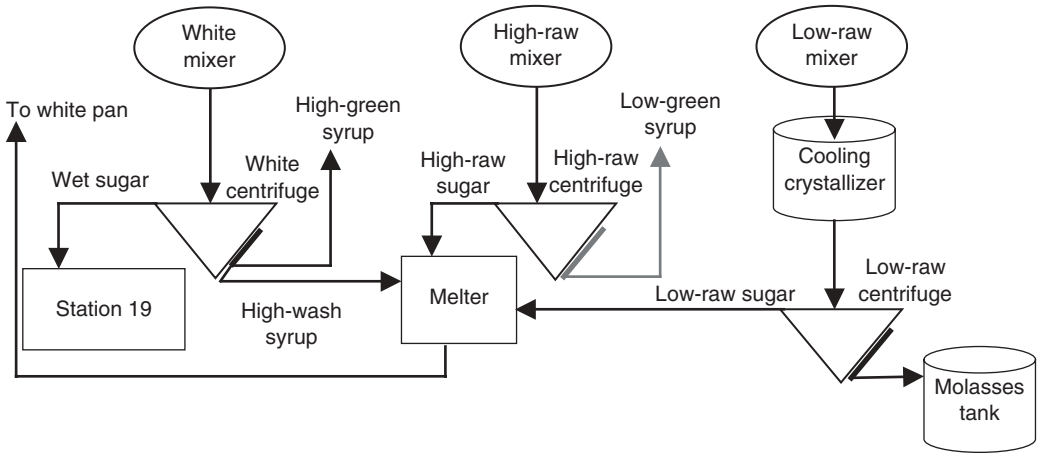


Figure 3.18.1 Flow diagram of centrifuging (Station 18)

The centrifugal station in the sugar factory separates the sugar crystals from the **mother liquor** (liquid around the crystals) in the massecuite by using centrifuges. A **centrifuge** is a machine that separates solid from liquid by **centrifugal force** (F_C), which is produced by high-speed rotation (about 1200 revolution per minute, RPM). The F_C pushes the mother liquor through the screen of the basket. Sugar crystals are too large to pass through and retain on the screen. During centrifuging, the mother-liquor components stay intact because the centrifugal operating has no effect on solutions. Centrifuges with a higher F_C can handle more concentrated massecuites.

FACTORS AFFECTING CENTRIFUGING

In the **centrifugal operation**, the following factors are important:

- Centrifugal force
- Massecuite temperature
- Massecuite concentration

The **centrifugal force** (F_C) pushes the rotating material from the center of rotation to the side. F_C is developed by rotation (*the faster the spinning, the greater the* F_C). F_C is given as

$$F_C = m\frac{v^2}{r} = m\frac{v^2}{D/2} = m\frac{2v^2}{D} \tag{3.18.1}$$

m	Mass of the spinning material
v	Velocity
r	Basket radius
D	Basket diameter

The velocity (speed) of the basket per 60 seconds is given as

$$v = \frac{\pi Dn}{60} \tag{3.18.2}$$

n Number of revolution per minute

Substituting v into (Eq.3.18.1) gives

$$F_C = m\frac{2(\pi Dn/60)^2}{D} = m\frac{2\pi^2 D^2 n^2}{3600D} = \frac{mD\pi^2 n^2}{1800} \tag{3.18.2}$$

The **gravitational force** (F_G = 9.81 m/s^2) is numerically almost equal to π^2= 9.86, so can be substituted for F_G in Eq. (3.18.2). The ratio of F_C to F_G is called the **separation factor** (Z):

$$Z = \frac{F_C}{F_G} = \frac{mD\pi^2 n^2}{1800\pi^2} = \frac{mDn^2}{1800} \tag{3.18.3}$$

Separation factor (Z) expresses the separating power of a centrifuge. This means that a centrifuge develops a F_C, which exceeds the F_G (see the Glossary) by the Z. Study the following example.

EXAMPLE 1

Given:

Mass of massecuite	1 t
Basket diameter	1.27 m (50 in.)
Revolution per minute (n)	1100

Calculate:
Separation factor (Z)
Cycle time

$$Z = \frac{mDn^2}{1800} = \frac{1 \times 1.27 \times 1100^2}{1800} = 854$$

This means that 1 t of massecuite is under a centrifugal force 854 times greater than the gravitational force (force of Earth's gravity), so the separation time of the mother liquor is 854 times shorter compared with a noncentrifugal operation (separation of the mother liquor without centrifuging). As shown in Figure 3.18.2, a Z of this magnitude shortens the separation time dramatically (*from a few days to a few minutes*).

Assume that the noncentrifugal operation takes 50 hours (3000 minutes). Then, the cycle time should be 854 times shorter. Therefore, the cycle time (in minutes) of this centrifuge is

$$\frac{3000}{854} = 3.5$$

CENTRIFUGING OF WHITE MASSECUITE

White sugar crystals are separated from the mother liquor by **batch centrifuges** and by washing with water during rotation to eliminate traces of impurities and to reduce sugar color (see Figure 3.18.3). **Continuous centrifuges** are usually used for high-raw (B) and low-raw (C) massecuites (see the following Note).

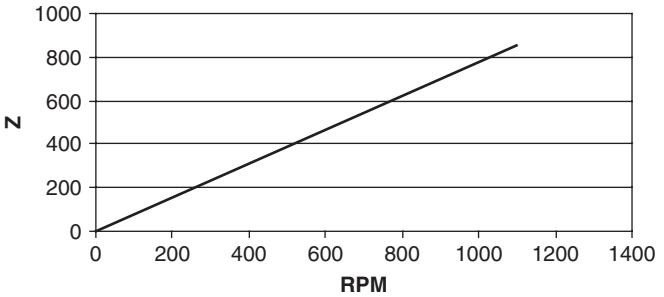


Figure 3.18.2 Relation between separation factor (*Z*) and speed of rotation (RPM)



Figure 3.18.3 Batch centrifuges for white massecuite centrifuging (BMA, K 2300)

White massecuite is centrifuged directly after it is discharged from the pan. When crystallization is complete, the massecuite from the pan is discharged into the **mixer** (a cylindrical tank with a mixing device) to wait for the centrifuging process. When the loading step of the centrifuge starts, the gate of the mixer is opened, and the massecuite is discharged into the centrifuge. In the centrifuge, the massecuite is rotated in the centrifuge basket, which has a perforated wall covered with a metal screen. Because of the centrifugal force, the mother liquor is removed from the sugar crystals and flows through the basket screen (see Figure 3.18.4).

Among other factors that influence its performance, the centrifuge's functionality depends on the massecuite *DS*, so the operating cycle must be adjusted accordingly. The operating cycle of a batch machine consists of the following steps:

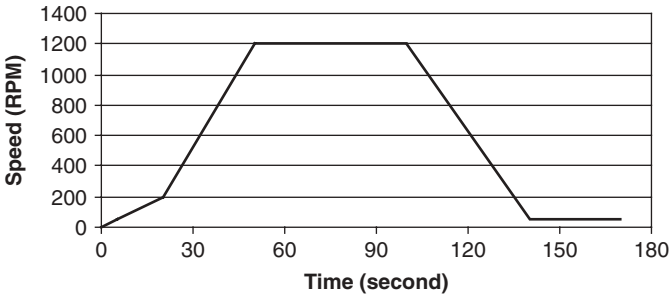


Figure 3.18.4 Centrifuge cycle steps

- **Charge:** The basket receives the massecuite through a chute while the basket rotates at a slow speed.
- **Accelerating:** RPM increases constantly to a predetermined speed to increase the F_C, which is evenly distributed over the entire height of the basket. F_C pushes the massecuite toward the basket's wall, discharging the mother liquor through the basket screen. The sugar crystals are too large to pass through and build up a layer 150 to 180 mm (6 to 7 in.) thick on the basket. In the meantime, most of the mother liquor, known as **high-green** (A-green) **syrup**, is removed. However, the crystals still contain a thin layer of mother liquor and appear yellow, so the crystals must be washed.
- **Washing:** The crystals are sprayed for a few seconds with hot water or steam, while the basket rotates at maximum RPM. During washing, the spray nozzle is moved up and down to wash the crystals uniformly. The runoff syrup discharged during this period is directed to a separate tank. This syrup is called **wash** (A-wash) **syrup** and has a higher purity than high-green syrup because part of the crystals is dissolved during washing. Constant rotation continues until the preset mass (or volume) of sugar crystals is reached.
- **Drying:** More liquor and wash water pass through the basket to achieve the desired moisture content (about 0.3%).
- **Braking:** the rotation is slowed and mechanical brakes stop the basket.
- **Discharge:** RPM decreases to discharge speed, the discharging device opens to discharge the crystals from the machine. A **plow** (blade) removes all remainders of the crystals from the basket. The wet sugar falls onto a screw conveyor, which moves the sugar to an elevator to feed the sugar dryer. At this time, the centrifuge is ready to start the next cycle.

Typical cycle times of a batch centrifuge are shown in the following table:

Cycle	Time (seconds)	Speed (RPM)
Charge	20	200
Accelerating	30	1200
Spinning	50	1200
Braking	40	50
Discharge	30	50
Total	170	

NOTE

Several suppliers (e.g., Broadbent) have developed continuous centrifuges for processing white massecuite. At this time, the use of continuous centrifuges has not been accepted by the beet-sugar industry because they produce sugar with relatively high moisture content (about 1.5% compared with 0.3% with batch centrifuges). However, technologists expect the use of continuous centrifuges for the white side to be improved. These centrifuges are similar to continuous centrifuges used for raw massecuites except they have, among other differences, a lower centrifuging force (Grimwood et al. 2000).

Sugar crystals are washed because of the following reasons:

- Increase the purity of the sugar
- Decrease the ash content of the sugar
- Decrease the color of the sugar

For washing, about 80°C **wash water** is used. Some factories use low-pressure steam at about 110°C. Most of the steam is condensed before reaching the crystals. The amount of wash water used depends on the type of massecuite, the sugar color, and the ash content requirements of the white sugar. White crystals are usually washed twice, which increases the purity of the sugar and decreases its color. The first wash is 2 to 4 seconds, and the second wash is shorter. There is an interval of a few seconds between first and second wash.

Wash water consumption is important (*the lower, the better at a given color reduction*). Wash water (including screen washing) used in white massecuite is 2 to 3% per weight of massecuite (see the following Note). Water washes off the colorants on the surface of crystals but not those colorants occluded in the crystals. Some sugar technologists express the washing process according to its time cycle (e.g, a two-second first wash).

The following should be practiced for efficient washing:

- **Correct spray nozzle:** Use triplet (three sets) or quintuplet (five sets)
- **Correct water distribution:** Use water equally over crystals
- **Quality water:** Use steam or condensate without ammonia
- **Correct water pressure:** Use pressure about 5 bar (73 psi)
- **Correct water temperature:** Use water 80 to 110°C

Steam has the following advantages over water for washing:

- **Moisture content:** Sugar discharged from the centrifuge contains less moisture (about 0.3%) than with hot water. This is because condensate formed from steam is removed more easily by centrifuging, so steam washing during centrifuging helps the drying of sugar in the dryer.
- **Temperature:** When steam is used, the sugar has higher temperature at discharge, so higher temperature helps the drying of the sugar.
- **Crystal hardening:** When hot water is used, the sugar might dry in the upper part of the basket, which may create problems when the sugar is discharged.

In general, coarse crystals are easier to centrifuge, creating a better yield. Very fine crystals pass easily through the centrifuge screen and become part of the mother liquor, which has to be

crystallized again, reducing the yield. Sugar factories try to produce **medium-size sugar**, which is 0.35 to 0.40 mm (350 to 400 microns or simply 350 to 400 *MA*) in size.

NOTES

- **The amount of wash water used during centrifuging is important to sugar loss and the energy use of the factory. A 1200 kg batch centrifuge uses 36 kg of wash water (at 3% wash water on massecuite), which exits the machine with the mother liquor. Later, in the pan crystallizer, this water is evaporated, which requires some heat energy (about 10 kWh per ton of massecuite). Therefore, wash water should be used with caution.**
- **Ash reduction by maintaining the wash time during centrifuging is important. You must maintain the washing time to produce sugar with an ash level below 0.015%.**

CENTRIFUGING OF RAW MASSECUITES

Today, most factories use **continuous centrifuges** for raw massecuites (high-raw and low-raw massecuite). Figure 3.18.5 shows a continuous centrifuge. The viscosity of the raw massecuites high-raw and low-raw massecuites) and their mother liquor is much greater than that of white massecuite. Therefore, the separation efficiency of mother liquor from the massecuite is greatly affected by the viscosity of the massecuite, the quality (the purity and color) of the low-raw

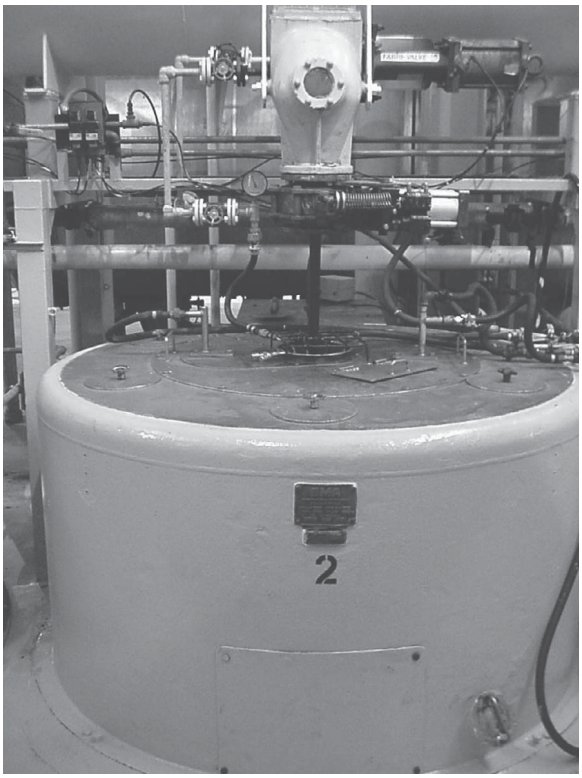


Figure 3.18.5 Continuous centrifuge

sugar, and, consequently, the melting process. Modern centrifuges can tolerate high-viscosity massecuites efficiently to produce high-quality sugar.

The exhaustibility of mother liquor is important in an efficient centrifugal operation. It is essential to have the mother liquor as close to saturation as possible before centrifuging. Maintaining supersaturation of the mother liquor to about 1 produces the best massecuite viscosity. Keeping supersaturation and viscosity at their optimum creates the best separation during centrifuging at the lowest possible water usage and sugar loss.

The centrifugal cycle (if batch centrifuges are used) of low-raw massecuite is two to four times longer than that of white centrifuges. In general, coarse crystals are easier to centrifuge, creating a better yield. Very fine crystals easily pass the centrifuge screen and become part of the mother liquor, which has to be boiled again.

For low-side centrifuging, washing is usually not used, or low washing is practiced. After centrifuging, the high-raw and low-raw sugars are directed to a screw conveyor to be transported to the melter. There, the raw sugars are dissolved in the thick juice to produce standard liquor, which is used as feed for the white pan.

DOUBLE CENTRIFUGING

Double centrifuging was offered in the 1980s to cover the following deficiencies of single centrifuging:

- Inefficient centrifuging
- Loss of some sugar to the mother liquor
- Difficulty determining the timing of washing
- Difficulty controlling the separation of green from wash syrup

In double centrifuging, the massecuite is first precentrifuged without being washed. Usually, the liquor separated in the precentrifuging step is returned to the massecuite just before the massecuite reaches the centrifuge. The liquor-return operation has an effect similar to that in the **molasses-recycle** operation (see Station 17), except that the massecuite flow through the remainder of the process is reduced rather than increased. As a result, the sugar loss to the mother liquor is decreased (lower mother liquor purity than in single centrifuging). Double centrifuging can be provided with single or double centrifuges.

Precentrifuging schemes vary significantly. One of the most interesting is the Beghin-Say Eridania factory in Abbeville, France, which uses some of the most modern continuous crystallization equipment available today. In this operation, a portion of the low-raw massecuite is sent to the precentrifugal section, and the balance of the massecuite is sent to the cooling crystallizer. The syrup from the crystals in the precentrifuging is combined with the massecuite feeding the cooling crystallizer, while the sugar is sent either to affination (see Station 17 for more information about affination) or directly to the melter. This scheme creates a purity drop of about 20 units in raw-side operation and production of molasses with about 60% purity (Rhoten 2004).

TYPES OF CENTRIFUGES

The **sugar industry** uses top-suspended centrifuges. From a design point of view, the batch centrifuges used for centrifuging white, high-raw, and low-raw massecuite are the same. However, the speed of rotation and basket size are different for individual stages.

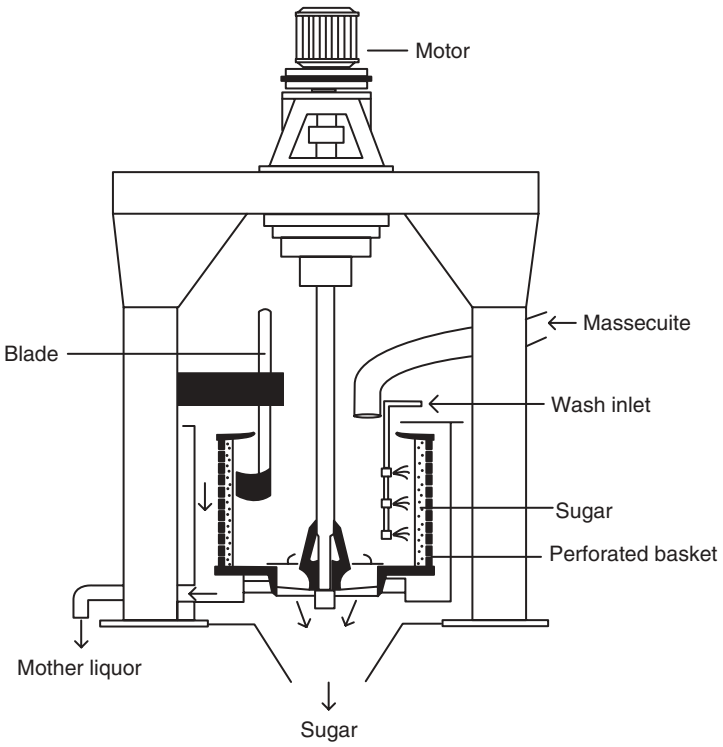


Figure 3.18.6 A typical batch centrifuge

Batch Centrifuge

In its **typical design**, a batch centrifuge (shown in Figure 3.18.6) contains a basket 1000 to 1370 mm (40 to 54 in.) in diameter and 700 to 1000 mm (28 to 39 in.) high. The wall of the basket is at an angle of about 30°. The ratio of height to diameter is usually below 0.8. The basket is perforated and lined with a stainless-steel screen, which functions as a filtering media. The basket is connected to a free-swinging vertical shaft driven from above by a motor. Different centrifuge suppliers use different types of motors, drives, and inverters. The speed of rotation of the basket ranges from 850 to 1800 RPM. To reduce vibration, the baskets are suspension-system designed with some cushion pads to absorb vibration.

An adjustable unloading blade inside the basket moves inward and downward to remove the crystal layer accumulated on the basket wall and then returns to its original position. The discharging device is actuated by a direct or an indirect drive. Three or five sets of spray nozzles are on a vertical pipe for uniform washing of the sugar crystals. The discharging valve of most centrifuges opens downward (in BMA and Western States centrifuges). In the closed position, the discharging valve fits tight to prevent air being drawn into the basket.

New centrifuges are programmable controlled to ensure accurate RPMs in different operating cycles. The total operating cycle per charge depends on the centrifugibility of the massecuite. White operations need shorter cycles (2 to 3 minutes) than raw massecuites.

The loading capacity of older centrifuges is 1000 to 1200 kg per cycle; new models can centrifuge 1750 to 2200 kg of massecuite. At a massecuite crystal content of 50%, for example,

a machine centrifuging 2200 kg of massecuite can produce 1100 kg sugar in one cycle or about 420 t per day. (Note: The utilization rate is around 80% because real basket loads are lower than nominal loads.)

Some newer centrifuges are equipped with a frequency-regulated drive that can adjust the speed of the basket to lower energy requirement. Some centrifuge suppliers use inverter drives instead of multi-speed drives in large batch centrifuges (above 2200 kg capacity). Centrifuges with inverter drives are more energy efficient than multi-speed drives (Grimwood 1999). However, the centrifugal station has a high power demand (40 to 50% of the factory's requirement). The energy consumption of centrifuges is between 1.0 and 1.5 kWh per ton of massecuite.

Continuous Centrifuge

In **continuous centrifuges**, the basket rotates at a constant revolution and receives the feed without interruption. Feed enters the small end of a funnel. The funnel minimizes crystal breakage, thus distributing the massecuite smoothly. Massecuite enters at the axis of rotation of the basket. Constant rotation and feeding creates an equal centrifuging force, causing uniform distribution of the massecuite. The massecuite gains speed and moves up toward the large end of the basket. While the feed is moving, the mother liquor flows through the basket screen. A layer of sugar crystals 25 to 75 mm (1 to 3 in.) thick is formed. The sugar layer is moved up by a reciprocating pusher. Each stroke of the pusher moves the sugar toward the edge of the basket, from where it falls into a collector chute. Cycle timers and solenoid-operated valves control the operation. Washing during rotation is controlled as desired.

Continuous centrifuges offer higher efficiency than batch types because of the short cycle and no holdup for loading and unloading. However, continuous centrifuges require more energy than batch ones because the energy to accelerate the massecuite is not recovered (van der Poel et al. 1998). Batch-type centrifuges are not effective in white-massecuite centrifuging but are useful in high-raw and low-raw centrifuging. Continuous centrifuges are not used in white massecuite for a number of reasons:

- Breaking of crystals at the loading step
- High moisture content in the sugar
- Formation of crystal lumps

Crystals break and become damaged mainly because of the high-speed rotation of the basket during the loading step.

Double Centrifuge

A **double centrifuge** consists of two units, one installed on top of the other. The baskets are either on the same drive (Dorr-Oliver) or driven with individual drives (BMA). In both designs, the massecuite is first precentrifuged in the top unit and centrifuged again in the bottom unit with or without washing.

The disadvantages of double centrifuging are its high capital and maintenance costs. High-energy consumption (2.0 to 3.0 kWh per ton of massecuite compared with 1.0 to 1.5 in single centrifuging) is another disadvantage of double centrifuging.

Centrifugal Screen

The **centrifugal screen** (filter) is important part of each centrifuge. The screens come in woven wire mash, drilled type, and laser-generated type. The screen openings are different depending on the crystal size. Screens have about 20% open area. The sugar industry mainly uses backing layers made of stainless steel to improve the efficiency of the screening (filtration) operation.

The lifetime of a screen ranges from 100000 to 150000 cycles, but ammonia in the washing water lessens the lifetime of the screen because it attacks the metals.

Centrifugal Control System

Modern centrifuges have a high loading capacity (up to 2200 kg), powerful components, and an efficient drive. They are equipped with a programmable controller for their sensors and actors that can be managed from the centrifugal control room. The control systems can set effectively the cycle time in different steps of centrifugal cycle. The newest models even offer teleservice, which graphically displays operational data on the monitor (next to the machine or in the control room) for better operation optimization.

OPERATING PROBLEMS

A number of **problems** can occur in centrifuging operations.

Basket Vibration

When the basket vibrates, the rotation axis swings (oscillates), forcing the basket into a nonsymmetrical position. If this situation is not corrected, the machine can be damaged. Modern factories use electronic oscillation measurements to detect centrifugal vibration (van der Poel et al. 1998). Modern centrifuges are programmed and dynamically balanced for optimum performance at high RPM. However, the usual causes for vibration of the basket are the following:

- Bent spindle
- Clogged screen holes
- Slippage of the screen
- Crystals stuck on the basket wall
- Loading at high speed (above 300 RPM)
- Wrong load amount (too high or too low)
- Centrifuging of a massecuite with low crystal content

Mother Liquor Stays above Crystals

At the beginning of loading and washing, the mother liquor might stay above the sugar crystals for a very short period. If the situation persists, something is wrong. Possible causes of this problem are as follows:

- Nonuniform sugar crystals: When crystals are not uniform and the amount of very fine crystals is unusually high, the fine crystals are compacted between larger ones, leaving less empty space. This prevents the easy passage of the mother liquor through the crystals and the basket screen.

- Washing nozzles are improperly placed
- Low-concentrated massecuite
- High-speed rotation
- Presence of foam
- Improper screen

Unusual Screen Damage

These are a number of possible causes of unusual screen damage:

- Basket vibration
- Wrong screen installation
- High ammonia in the wash water
- High-speed rotation during discharging
- Bumping the blade to the screen while discharging

Low Centrifugal Efficiency

Following are possible causes of low centrifugal efficiency:

- Low charge
- Fine crystals
- Low massecuite temperature
- High-foam-content massecuite
- High-concentrated massecuite

Inconsistent Sugar Crystal Quality

The sugar crystals produced in one load should be of equal quality with respect to color and purity. If the sugar quality is inconsistent, it may be due to one of the following:

- Wash water over crystals is not equally distributed
- Low amount of washing water
- Low water pressure
- Dirty screen

Centrifuged Sugar is Too Wet

In white centrifuging, wet sugar discharged from the centrifuge should not have moisture content of above 0.3%. Sugar with high moisture content creates problems during transport to the wet box as well as in the box because it gets hard. The following are the reasons for centrifuged sugar with high moisture content:

- **Short drying period**: If the drying period of the cycles is short, enough mother liquor does not pass through the basket to achieve the desired low moisture content.
- **Crystals are too fine**: At the same separation factor (Z) of the centrifuge and viscosity of the mother liquor, extra fine crystals impede the separation of the mother liquor from the crystals, causing high moisture content in the centrifuged sugar.

- **Massecuite is too viscous**: Higher viscosity of the mother liquor impedes the release of the liquor from the crystals.
- **Massecuite is too concentrated**: White massecuite with high *DS* (higher than 93%) slows down the separation of the mother liquor from the crystals, which also causes high moisture content of the sugar.

NOTES

- **The product of the centrifugal station is wet sugar, which after drying becomes the product of the factory. Because the sugar is in direct contact with centrifuge components (e.g., the discharge blade and the spray nozzle), contamination can occur. Contaminants such as rust particles (mostly from the discharge blade or spray nozzle) can be separated from the sugar in a later stage of operation. Others such as hydraulic oil and grease from the discharge device of the machine cannot be corrected. Oil contamination is a serious problem with sugar quality and its source must be identified immediately. In the case of oil contamination, frequent sampling should be performed until the problem is identified and corrected.**
- **In the next section (Station 19), you will follow the operation of sugar production to its end, where the wet sugar leaving the centrifugal station is dried, screened, conditioned, and stored or sent for packing.**

CHAPTER 3

SECTION 19

SUGAR DRYING, STORING, AND PACKING

SUBSECTIONS

- Sugar Drying and Cooling
- Types of Dryers
- Sugar Screening
- Sugar Storing and Conditioning
- Sugar Silo
- Sugar Packing
- Packed-Sugar Storing
- Sugar Transport
- Safety Precautions
- Operating Problems

ABOUT THIS SECTION **This section** familiarizes the reader with **Station 19** (sugar drying, storing, and packing) of sugarbeet processing. Station 19 (see Figure 3.19.1) starts from the point when wet sugar from the centrifugal station (Station 18) enters the sugar dryer and ends when the sugar is in the silo, in the bag, or processed into the specialty sugars (Station 20).

The wet sugar leaving the centrifuge is first sent to the sugar dryer to reduce its moisture content. The sugar is then cooled and conditioned, during which the moisture content is lowered by flowing **conditioned air** (air with a certain temperature and humidity) to the desired level (about 0.03%). Conditioned sugar with a low-moisture content does not harden during storing and shipping in bulk form or in the bag. The sugar that passes through Station 19 is ready for the market or processed into specialty sugars such as powdered sugar, brown sugar, or cube sugar.

Beet-Sugar Handbook, by Mosen Asadi
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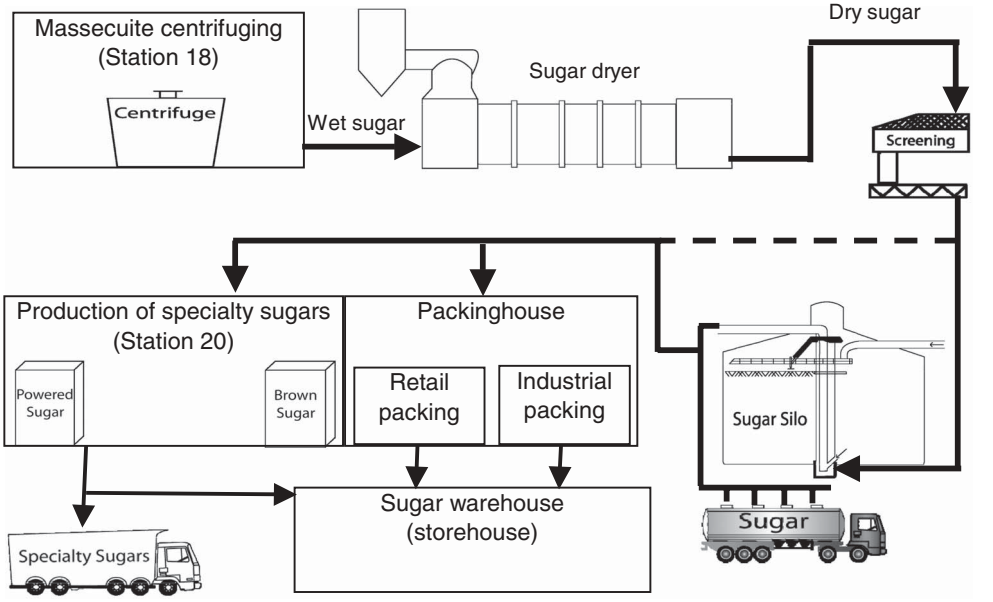


Figure 3.19.1 Flow diagram of sugar drying, storing, and packing (Station 19)

The **wet sugar** discharged from the centrifugal station has a moisture content. of 0.1 to 1% (depending on the amount of washing during centrifuging and the centrifuge's efficiency) and a temperature of about 60°C. The moisture is removed by drying in the sugar dryer.

SUGAR DRYING AND COOLING

Drying is the process of removing a liquid (usually water) from a wet solid substance by the use of heat (usually hot air). The concentration difference between the heating medium and the solid substance under drying is the **driving force** (cause) of drying, which evaporates the water in the substance.

Before starting to discuss the main subject, one important point must be made. **Today**, most sugar factories **dry** and **cool** granulated-refined (GR) sugar in the same equipment because of the high **heat content** (enthalpy) of wet sugar. The **enthalpy** present in wet sugar entering the **sugar dryer** (granulator) is usually sufficient to dry itself to the desired level without the need for additional heat. In normal operations, if the sugar entering the dryer has a moisture content below 1% and is at about 60°C, its heat provides sufficient energy to reduce the moisture to about 0.05%. Because hot air is not used, the sugar gradually cools to below 35°C when leaving the dryer. During transportation from the dryer to the silo or to the packing station, the sugar cools further to a satisfactory temperature of around 20°C. Therefore, the term **drying of sugar** in this section refers to both drying and cooling the sugar. All sugar dryers described in the following pages (except the rotary-tray dryer) function as dryer-coolers. (Note: Some sugar factories have a separate sugar cooler, for example, a fluidized-bed cooler, in which warm sugar cools by **conditioned air** [air with a certain temperature and humidity.])

The process of drying and cooling sugar consists of removing water from wet sugar to the desired level and cooling the sugar to the norm. As wet sugar dries, the **surface moisture** is actually evaporating from the sucrose solution around the crystals.

Most of the water in wet sugar is on the surface of the crystals in the form of a saturated sucrose solution with high purity (very close to 100%) and high concentration (about 80%). The wet sugar is dried and cooled in the sugar dryer to a moisture content of about 0.05% and a temperature below 35°C. The sugar loses more moisture and cools during transportation from the dryer to the silo or in the packing station. To ensure that the sugar is safe for packing and shipping, it is usually held for 24 to 72 hours (depending on its moisture content) under sufficient air current with low **relative humidity** (see the Glossary), e.g., in a sugar silo. During this process, known as **sugar conditioning**, the sugar reaches the equilibrium moisture content, and its temperature is reduced. The conditioned sugar has a moisture content of about 0.03, has an ambient temperature, and is **free flowing**. Sugar with such properties is safe for storing, shipping in bulk form, or shipping in a bag. (Note: The conditioned sugar is shiny compared to wet sugar, which looks dull.) There will be more details on sugar conditioning in the Sugar Storing and Conditioning section.

After the wet sugar is discharged from the centrifuges, a screw conveyor, belt conveyor, or bucket elevator moves it into the **wet sugar box** (bin). The wet box works as a hopper feeding the wet sugar to the sugar dryer. In the dryer, the heat of the wet sugar usually evaporates the water in the sugar. If the moisture content of the wet sugar is higher than normal (more than 1%), additional heat is necessary to dry the sugar.

Water exists in wet sugar in three forms:

- **Surface** (free) **moisture:** Exists on the crystal surface. The surface layer contains most of the water present in the crystal.
- **Interior** (bound) **moisture:** Exists near the surface of the crystal. It takes about 24 to 72 hours for the bound moisture to escape, depending on the properties of the conditioned air (flow rate, temperature, and relative humidity).
- **Inherent moisture:** Exists completely inside the crystals. The amount of inherent moisture is extremely small and is difficult to remove but does not create any problem in packing or storing of the sugar.

Most of the moisture in wet sugar is surface moisture, so the main part of the heat is used to vaporize surface water, which is easily removed in the dryer. Almost all the remaining moisture left in the crystals is bound moisture present inside crystals and is removed only over time. Therefore, the removal of bound moisture to a desired level (below 0.03%) continues during the cooling and conditioning of the sugar.

The drying of sugar crystals in the dryer can be studied considering the **drying rate** (R, the change of water content per unit time) in three different periods (see Figure 3.19.2):

- **Zero-rate period:** R is zero, establishing an equilibrium condition between the sugar crystals and the drying air.
- **Fast-rate period:** R is fast, creating a sharp downward curve.
- **Slow-rate period:** R is slow, creating a mild downward curve.

Let us see what happens during the drying of wet sugar crystals, which are covered by a layer of saturated sucrose solution. In the first part of the drying process, when the relative humidity

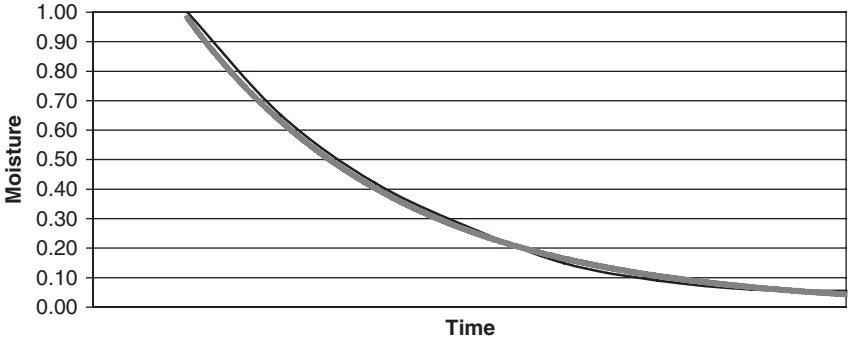


Figure 3.19.2 Typical drying curve for sugar crystals

(RH) corresponds to the concentration of the saturated solution, no drying takes place, but conditions for drying are established, creating a **zero- rate period**. As the drying proceeds, the RH in the air around the crystals is reduced, developing a **fast-rate period**. Towards the end, the movement of water from the solution around the crystal reduces, creating a **slow-rate period**. Generally, the drying process during the fast and slow periods is accompanied by a crystallization process. Crystallization occurs because the solution around the crystals becomes supersaturated as the water content decreases, so the solute (in our case, sucrose) moves from the supersaturated solution and attaches to the crystal structure. During the slow-rate period, the crystallization process becomes slower because of the increasing concentration and viscosity of the solution (*high viscosity slows the crystallization process because it acts as the opposing force of crystallization*).

As the drying process slows as the water content of the sugar becomes low, the drying time in the dryer is nevertheless maintained to dry the sugar to a certain moisture content (around 0.05%). As mentioned, reducing the moisture content further to the desired level (below 0.03%) occurs during the cooling and conditioning of the sugar.

In sugar drying, the optimum drying rate and consistency are important. In an **ideal sugar drying**, each sugar crystal goes through a cycle similar to that shown in Figure 3.19.2. The temperature of air entering the dryer is constant, but it changes as it moves in the dryer. High-temperature drying air creates **rapid drying**, which makes the surface of the crystals hard. As a result, the interior water cannot be easily removed during the cooling and conditioning period. This effect is known as **case hardening**. In addition, rapid drying makes the crystals look dull. On the other hand, **slow drying** (low-temperature drying air) can cause insufficient drying, creating hardening sugar in the bag and in the sugar silo. Note: Under equal conditions, the rate of drying depends on the crystal size (*the larger the crystal size, the smaller is the surface area and, therefore, the greater is the rate of drying*). Also, note that it is not always possible to find the optimum drying rate by calculation, so it must be found by experiment.

Usually, the sugar is cooled to 25 to 35°C, depending on the dryer-cooler availability in the factory and the temperature of the weather. Most factories have a drum or louver dryer with a cooling zone at the last section to cool the dried hot sugar. Other factories have a fluidized-bed dryer, whose last compartment works as a cooling section. Some factories have a separate sugar cooler (drum or fluidized-bed cooler) to cool the dry sugar to 25 to 30°C, which is a safe temperature for storing sugar in silo or packing.

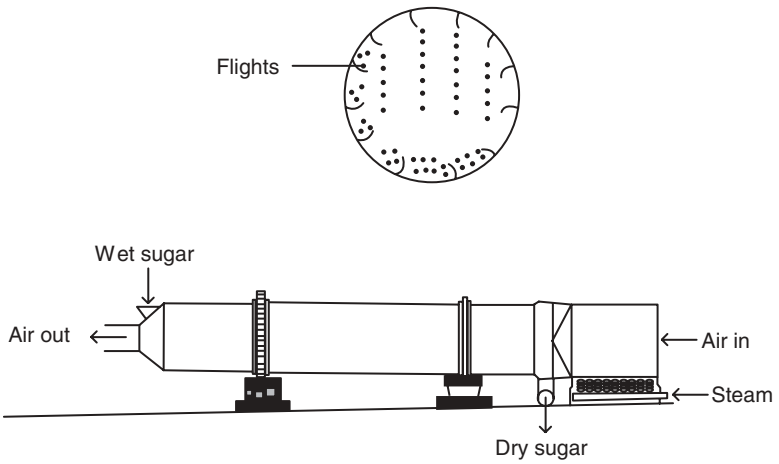


Figure 3.19.3 Rotary-drum dryer

TYPES OF DRYERS

The dryers used for sugar drying differ mainly in the way the sugar and the drying air are moved in the dryer. Four types of dryers are mainly used for sugar drying:

- Rotary-drum dryer
- Rotary-louver dryer
- Fluidized-bed dryer
- Rotary-tray dryer

Rotary-Drum Dryer-Cooler

In newer models of **rotary-drum dryers**, the sugar is dried and cooled in a single drum. A **rotary-drum dryer-cooler** consists of a rotating drum 2 to 3 m (6 to 10 ft) in diameter and 15 to 20 m (45 to 60 ft) long. As shown in Figure 3.19.3, the dryer is slightly inclined toward the outlet. The wet sugar enters the upper end of the dryer, and the dry sugar is discharged from the lower end. The sugar in the dryer is moved by the incline of the drum and the **flights** (arms for lifting a substance), which are mounted to the drum and rotate with it. The dryer is supported on two sets of bearer rollers and driven by a gear and pinion. The sugar enters the feed chute at the upper end, passes the air-discharge hood, and enters the dryer. Air enters at the lower end by the force of the fan, and exits the dryer from the upper end. Air can be heated by going through the air heater. At the lower end, the dry sugar is discharged into a conveyer.

As the drum rotates, the flights lift the sugar crystals and shower them down through the current of air. The sugar is lifted and falls several times during one revolution of the drum, to ensure better drying. To reduce moisture to the safe level when the weather is cold or when the wet sugar temperature is below 60°C and contains more than 1% moisture, air goes through the air heater. Otherwise, the sugar supplies the heat because sugar leaving the centrifuges is still warm. Air is always filtered before entering the dryer.

NOTE

Old rotary-drum dryer-coolers consist of two drums: one drum is used for drying and the other for cooling. The sugar is moved from the drying drum to the cooling drum through a series of chutes. Air passes over steam-heated coils to be heated to about 100°C (232°F) before entering the drying drum. The air entering the cooling drum is not heated. The drying and cooling air are drawn together from the middle of the dryer. These dryers are not built anymore because of their low heat efficiency, higher cost, and larger floor-space requirement.

Rotary-Louver Dryer-Cooler

Rotary-louver dryers (rotary-screen or roto-louver dryers) are widely used in the United States. In a rotary-louver dryer (see Figure 3.19.4), sugar crystals are in partial fluidization by the action of the louvers mounted on a rotating drum (Brocklesby 1998). Air passes a heater to give the optimum conditions for drying and then is forced by a fan into the dryer. In the dryer, the air moves upward at about 0.5 m per second through the screen and then through the sugar, leaving at the discharge end. The countercurrent movement of the sugar and air in the dryer prevents the dry sugar dust from mixing with the wet crystals. The dried and cooled sugar leaves the discharge end at an inclined angle to the air discharge.

Fluidized-Bed Dryer-Cooler

Fluidized-bed dryer-coolers (see Figure 3.19.5) have been used in the sugar industry since the 1980s. They were used first to cool sugar and later for both drying and cooling sugar. Air moves faster than in other dryers at about 1 m/s to keep the sugar crystals in **full fluidization**. Because the sugar crystals in the dryer are in constant motion by the velocity of the drying air, they behave like a fluid being showered through the air, so the process is known as **fluidization**.

Fluidized-bed dryers used in sugar factories have two separate compartments through which the sugar moves from the drying compartment to the cooling compartment. The retention time is short (about 30 seconds) and is almost the same for all particles. Drying conditions can be changed from one compartment to the other, and the second compartment is supplied with unheated air to cool the sugar before it is discharged. BMA and Stork fluidized-bed dryers are the most common fluidized-bed dryers in the sugar industry.

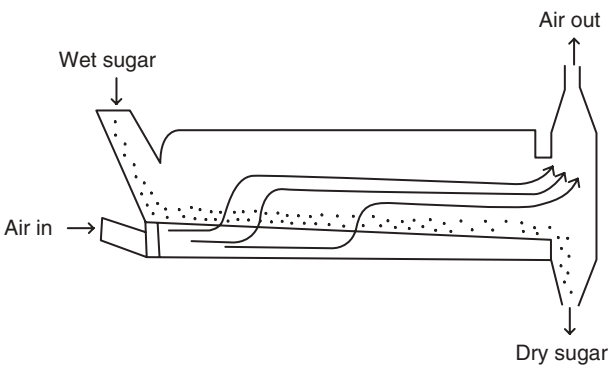


Figure 3.19.4 Louver-conveyor dryer

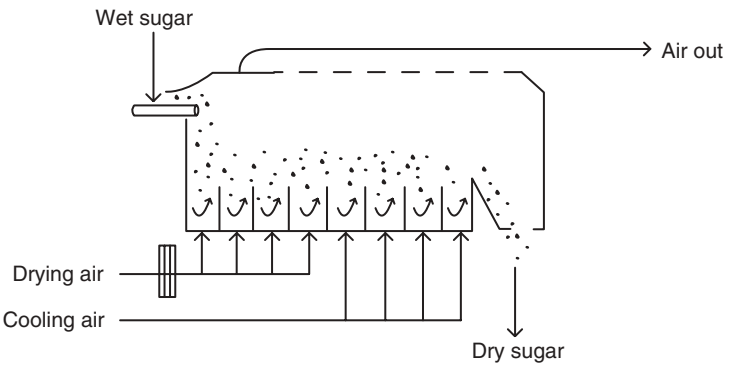


Figure 3.19.5 Fluidized-bed dryer

NOTE

To increase cooling efficiency and sugar input, some modern factories have modified their sugar drying-cooling system by using their old equipment as only a dryer and installing a separate fluidized-bed cooler. Sugar with a temperature of about 55°C leaves the dryer and enters the cooler, where is cooled to 25 to 30°C, which is a safe temperature for storing the sugar in a silo or for packing. In the BMA fluidized-bed cooler, the air leaving the system is filtered to remove dust and is then used to preheat the ambient air to an allowable temperature for entry into the cooler. Such a modification can increase the input of the sugar-end about 1.5 times.

Rotary-Tray Dryer

Rotary-tray dryers are used only to dry the sugar and are not popular in the sugar industry. This type of dryer has a vertical drum consisting of several horizontal trays, each with a slot. The trays are mounted on a vertical axis that rotates slowly. A scraper scrapes the sugar toward the slot, where it falls down onto the tray below. A blower circulates heated air between the trays.

These dryers are not well accepted because of the following reasons (Asadi 1978):

- Poor contact between the sugar and the air
- High damage to sugar crystals
- Low drying efficiency
- High energy demand
- High retention time

Comparison of Dryers

The most important point to consider in selecting a dryer is whether it dries and cools the wet sugar to the desired level at the lowest possible energy usage. Other important factors are the capital requirement, operating costs (mainly energy requirement), and ease of operability.

Both rotary (drum and louver-type) and fluidized-bed dryers can produce sugar with the desired moisture content and temperature. In both systems, airflow of different temperatures can be directed to the drying and cooling sections, allowing the drying and cooling of wet

sugar with different moisture contents and temperatures. Given the same moisture content in the wet sugar, we can make the following comparisons of both types of dryers:

- A rotary dryer needs less energy than a fluidized-bed dryer (because it needs less airflow)
- A rotary dryer is less dependent on the moisture content of the sugar than a fluidized dryer
- A rotary dryer requires partial fluidization but a fluidized dryer needs a full fluidization
- A rotary dryer needs less airflow (about 0.5 m/s) than a fluidized dryer (about 1 m/s)
- A rotary dryer breaks agglomerates better than a fluidized-bed dryer
- A rotary dryer is easier to operate than a fluidized-bed dryer
- A fluidized-bed dryer is less robust than a rotary dryer
- A fluidized-bed dryer damages the sugar less than a rotary dryer
- A fluidized-bed dryer occupies less floor space than a rotary dryer
- A fluidized-bed dryer removes sugar dust better than a rotary dryer
- A fluidized-bed dryer requires less maintenance than a rotary dryer
- A fluidized-bed dryer can separate fine particles better than a rotary dryer
- A fluidized-bed dryer requires shorter retention time (about 5 minutes) than a rotary dryer

Both types of dryers offer efficient operation with some advantages and disadvantages. The final decision about which one to choose depends on the capital requirement. When comparing the dryers as to capital requirement, however, take into account the incorporation of the dryer installation into the existing process, not just the dryer itself. For example, a factory that already has a rotary dryer and wants to expand its sugar-end operation can use the existing dryer for drying and install a fluidized-bed system to cool and condition the dried-warm sugar. This type of expansion requires less investment than to eliminate the old dryer and install a new one.

SUGAR SCREENING

The **screening** of sugar is the process of passing the sugar through a vibrating screen to separate undersized and oversized particles of sugar. The size of the screen to be used depends on need and on the desired size of product. Screening is performed after drying and after storing in some factories, when the sugar has to be shipped. The objectives of screening sugar are

- To screen sugar for producing the desired size crystals
- To separate very fine particles of sugar
- To separate the lumps formed during centrifuging and drying

A **screening machine** (e.g., Tyler or Rhewum types) contains an inclined screen that vibrates to pass undersized particles through the screen openings. In typical screening equipment, the screen sits on a frame with a 35 to 40° angle (see Figure 3.19.6). The sugar is fed at the higher end of the screen and spread over the entire screen surface, creating a thin layer of crystals. The incline of the screen and its rate of vibration determine the speed (rate) at which the sugar moves towards the lower end. The screen is usually adjusted to move the sugar at a rate of 1.0 to 1.4 m per second.

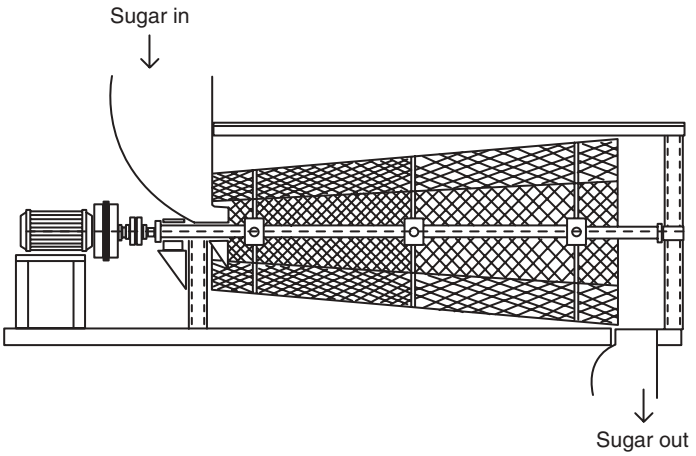


Figure 3.19.6 A typical sugar screen

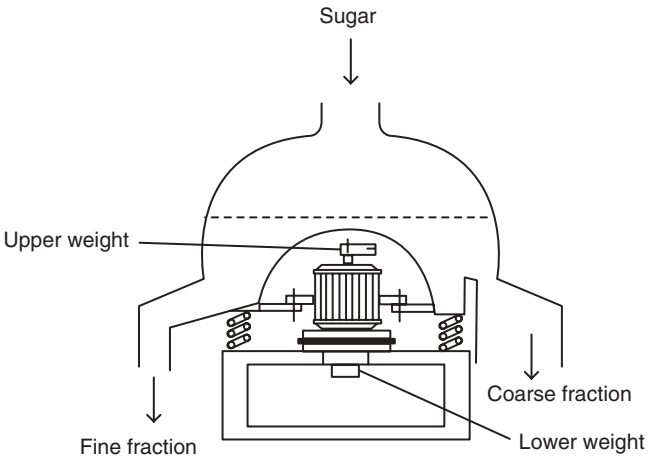


Figure 3.19.7 Sweco vibrating screen

Sifters (e.g., Rotex or Sweco types) are also used to screen sugar. The **Rotex** screening machine consists of two screens and operates by a rotating movement. The screens, made from a special cloth, sit on an inclined frame. Several balls are located under the screens and bump against the frame to prevent blocking of the screens. Every other day, the screens are replaced, cleaned by blowing air, and checked for possible holes.

The Sweco screen (see Figure 3.19.7) operates on a vibrating mechanism that is generated by two weights located at the upper and lower ends. The top weight creates a horizontal-vibrating movement and the lower weight causes a vertical-vibrating movement, both resulting in a two-direction movement.

In general, the following three factors should be considered for the effective operation of a screening machine:

- **The inclined angle of the screen:** The screen must be adjusted to achieve a suitable rate of movement of the crystals on the screen.
- **The speed of vibration:** The vibration must be adjusted to prevent fast movement of undersized particles because they cannot pass through the screen when the screen vibrates too fast.
- **The cleanness of the screen:** The screen must be kept clean to prevent blocking of the openings.

NOTES

- **Some factories are equipped with multiple-screen units to separate the sugar into three different sizes: fine, medium, and coarse. The fractions can also be blended together. A multiple-screen system needs sugar with a broader range of crystal sizes, so the crystallization station must operate in such a way to produce the necessary sizes of sugar crystals.**
- **The laboratory tests the crystal size using a benchtop screen instrument. The test uses five to seven screens, and the mesh size (the number of openings per surface unit) of the screens depends on the crystal size of the sugar produced by the factory. (See Chapter 11 for a discussion of the crystal size.)**
- **The mean aperture (*MA*) is the average crystal size of the sugar, expressed (in mm or μm) as the size of the screen openings on which 50% of the sugar sample stays and 50% passes through. The standard crystal sizes are as follows:**
 - **Extra-fine sugar, with an *MA* of about 200 μm**
 - **Fine sugar, with an *MA* of about 300 μm**
 - **Medium size sugar, with an *MA* of about 500 μm**
 - **Coarse sugar, with an *MA* of about 1000 μm**

SUGAR STORING AND CONDITIONING

After screening, the bulk sugar is **stored** in steel or concrete **silos**. The first **Weibull** steel silo was developed in Sweden in 1933. Since then, many sugar factories have installed steel silos with a capacity of 20000 to about 80000 t. The lower operating costs of bulk storing in a silo compared to storing in large bags (50 to 1000 kg) is the main reason for installing a sugar silo. (The ratio of labor cost is about 5 to 1 in favor of bulk storing.)

Sugar conditioning refers to holding sugar in a current of adequate air for a certain period after the sugar is discharged from the dryer so that the crystals can lose their bound water and reach the equilibrium moisture content. (Sufficient time is needed to move bound water out of the crystals.) If bound water is not removed and sugar is stored in a place without air ventilation, caking occurs. Therefore, sugar must be conditioned to a safe temperature (about 25°C) and moisture content (about 0.03%) in the cooler or in the silo with a complete ventilation system. The properties and the amount of airflow through the crystals are important. Conditioned air with 20 to 25°C and 55 to 60% relative humidity (RH) creates the best conditioning environment and corresponds to a sugar moisture-content of about 0.03%. In addition, the airflow rate is also important to achieve a complete conditioning operation and prevent condensation.

The time required for conditioning depends on the following:

- Size of sugar crystals
- Moisture content of the sugar crystals after drying
- The RH, temperature, and air current through the crystals

As stated, the sugar after drying still contains bound moisture that will be slowly released in the silo. A lack of suitable conditions in the silo to reduce the moisture content of the sugar to an optimum level (about 0.03%) causes the following problems:

- Increased crust formation on silo walls
- Increased color formation of the sugar
- Increased microbiological activities
- Decreased appearance of the sugar
- Decreased flowability of the sugar

The problems can be prevented by a good conditioning operation. Some factories have a separate sugar cooler (drum or fluidized-bed cooler) to reduce the moisture content of the sugar to about 0.03% and cool the dry sugar to 25 to 30°C, which are safe conditions for the storage of sugar in silos or packing. Some factories have a small **conditioning silo** (bin) for treating the sugar before storing in the main silo. Conditioning silos usually have a capacity equal to a retention time of 24 to 72 hours. In the bin, conditioned air (filtered air 20 to 25°C and with 55 to 60% RH) is continuously recirculated in the silo to decrease the moisture content of the sugar to a suitable level and remove the released moisture from the silo. The dust is removed from the silo to reduce the risk of **explosion**. The airflow rate is also important. Sugar technologists recommend an airflow rate of about 3 m^3 per hour per ton of sugar in the silo (Bruijn 1982).

SUGAR SILO

The sugar silo is for long-term sugar storage. Sugar silos have a concrete floor and steel or concrete walls. The most advanced types are wall-heated and insulated ones to prevent condensation.

These silos are ventilated with conditioned air to condition the sugar and create a uniform temperature profile across the silo. They have a dust-collecting system that collects and filters the generated dust to prevent fire explosion.

In general, a sugar silo consists of three stories. The first story is below ground level and contains the conveyor for transporting the sugar from the main building of the factory and the elevator. The second, or main floor, is for sugar storing. The third floor contains the conveying equipment for receiving sugar from the elevator and its delivery to the storage area or to a belt conveyor to transport the sugar out of the silo. Modern silos are equipped with a **ventilation system** with conditioned air 20 to 30°C and with a low RH. In these silos, sugar is continuously ventilated with conditioned air (heated or cooled air, filtered to remove dust) to prevent condensation. Under these storage conditions, sugar with a moisture content up to 0.05% is in equilibrium, so no caking and hardening or excessive surface moisture occurs. A few days after filling and when the sugar is conditioned, the ventilation air period is reduced to 2 to 5 hours a day.

A typical Weibull silo (see Figures 3.19.8 and 3.19.9) is a cylindrical steel tank 30 to 50 m (75 to 150 ft) in diameter with a capacity of 20000 to 80000 t sugar. The silo is ventilated and insulated, which is covered with aluminum sheeting. The sugar conveyor and an elevator for personnel are installed in a column. The column is mounted in the center of the silo and is about

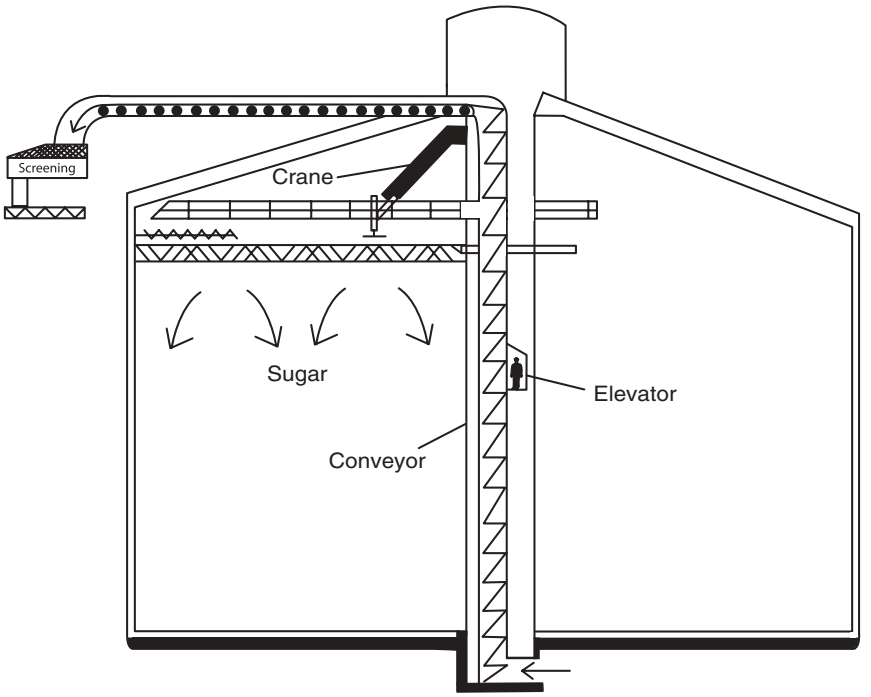


Figure 3.19.8 Design of a typical Weibull sugar silo



Figure 3.19.9 Weibull sugar silos (Courtesy, Michigan Sugar Company)

4 m (12 ft) in diameter. The conveyor takes the sugar to the top of the silo and feeds it to a distributing screw conveyor, which is carried by a crane (bridge). The crane rotates around the center column and can be raised or lowered automatically over the entire height of the silo. The sugar from the distributing conveyor is fed to a discharge chute and then falls over the storing area. The sugar is then spread evenly by an auger. During discharge, the distributing screw conveyor rotates in the opposite direction, and a belt conveyor transports the sugar out of the silo.

The temperature and RH of the **circulation air** in the walls, ceiling, and through the stored sugar are closely controlled to remove water released from the sugar during storage. After filling the silo and the conditioning period, the ventilation system can operate periodically 1 to 3 hours a day. During intercampaign, called **quiet period**, a discontinuous air-circulation process can be practiced.

NOTE

Several silo-suppliers in the world offer special silos for the storage of sugar, such as Weibull (Sweden), Abay-ABR [Belgium–licensed to iedeman (Germany)], Lucks (Germany), Niro-DDS (Denmark), ACMB (France), and Spaans (Holland).

SUGAR PACKING

The sugar packinghouse operates year-round receiving the sugar from the silo for producing different sizes of sugar packs. Granulated-refined (GR) sugar is packed in different sizes from a 5 g (teaspoon) bag, called a **packet** (for use in restaurants and other commercial places), up to 1000 kg (in the United States, 2500 lb). In general, sugar packs (packages) are divided into two sizes:

- **Industrial size:** Packs more than 10 kg (25 lb)
- **Retail size:** Packs up to 10 kg

Typically, the U.S. sugar-factories' sales by volume are 70% industrial and 30% retail size. The **retail** (household or consumer) packinghouse consists of a number of automatic packing machines. Usually, one or a few packing machines are used for packing particular weight packs. The most common sizes are 5 lb and 10 lb in the United States and 2 kg and 2.5 kg in other parts of the world.

Packing machines vary in design and speed. Some machines (e.g., the machines for producing packets) make their own packs from paper-roll stock; others (such as machines for producing 1 and 5 kg packs) use premade packs. All machines are fed by an overhead belt conveyor.

The machine for 1 and 5 kg packing line-packs 30 to 100 bags per minute, depending on the number of scales and dispensers. Modern machines, such as Sealtile from the United States and Hesser (Bosch) from Germany, have 12 scales and can pack up to about 220 packs per minute.

When packing **small-size bags**, the machine weighs the required amount of sugar and puts it into the packer, which packs the sugar into a two-walled paper bag. The bag is sealed with a **polyvinyl-acetate** (PVA) glue, folded over, and compressed to set the glue. The closed bags are placed inside a moving housing. Packs travel to a **baling** machine, which puts six or

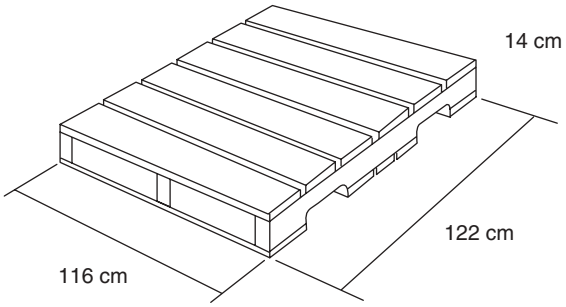


Figure 3.19.10 Wooden packing pallet

twelve packs (depending on pack size) inside Kraft paper (see the following Note) to produce a **bale** (bundle) of packs. The paper for baling can be purchased or made directly on line. The bundle is closed, glued, and palletized. Many factories have automatic **palletizing machines**, which take the bale from the production line and stack them on a wooden **packing pallet**. A typical size packing pallet is 122 × 116 cm (48 × 40 in.) wide and 14 cm (5.5 in.) high, as shown in Figure 3.19.10.

Common industrial-size packs are 20 kg (50 lb in the United States), 40 kg (100 lb), 100 kg, and 1000 kg (2500 lb). Bags for packs up to 50 kg are made of three-walled paper. Unlike small bags, large-size bags are not packed in bales but are instead stacked directly on the pallet. These bags are purchased from suppliers.

Sugar is also packed in **super bags**, called **flexible intermediate bulk containers** (FIBC). Super bags are popular in the United States and are referred to as **jumbo bags**, **tote bags**, and **super sacks**. These bags range from 1500 lb to 2500 lb (see Figure 3.19.11). Super bags are made of woven polypropylene and lined with low-density polyethylene liner that provides a moisture barrier.

Super bags are filled through a top spout. The fill spout is fitted around the discharge spout of the filling machine. An air device on the discharge spout of the filling machine inflates to seal the tote fill spout and holds the tote in place to prevent sugar dust from escaping. The inner liners are sealed with a plastic cinch-down seals. The fill spout of the super bags is equipped with a sewn-in string that is tied around the spout to complete the closure.

NOTES

- **Kraft papers are the most common packing material for bags. The weight of the paper used in retail (consumer) packs is generally 100 g/m^2. The bags are made of a two-walled paper to provide strength. The outer wall is white or colored and the inner wall is light brown.**
- **Premade bags in sugar packing are supplied by paper-bag suppliers. The bags come in flat-folded form and open easily by the packing machines.**
- **Weight control of packed products is explained in Chapter 4 and in Example 38 in Chapter 12.**



Figure 3.19.11 A super bag for a 1000 kg package sugar pack

PACKED-SUGAR STORING

Packed sugars on pallets from the packinghouse are moved by lift trucks to the **sugar warehouse** (storehouse) and stacked in piles from 10 to 15 m high. To allow a passage way and air circulation, aisles of about 0.75 m are left between each piles and between the piles and the walls. Such an arranged warehouse can store about 0.5 t of sugar for each m^3 of space. The sugar warehouse is ventilated continuously to keep the humidity under control. In cold weather, the warehouse is heated with warm air to prevent condensation and hardening the sugar in the bags.

Packed sugars on pallets are transported to the customer mostly by truck or by railcar in some area. Palletized shipments are used to deliver sugar to the warehouses of grocery stores and to some industrial customers who use the sugar as an ingredient in their products. The industrial customers that have **bin** (small silo) prefer the sugar be shipped in bulk.

SUGAR TRANSPORT

Today, more than half the sugar is transported in bulk form by tankers or railcars. **Mobile bins** are also used to deliver sugar to customers who do not have their own bin and need large loads of sugar.

Most sugar factories contain one or two bins for holding bulk sugar after it is discharged from the silo and while it is waiting to going through the sifters for screening to the desired size. Sugar of an undesired size is returned to the silo, and the dust is directed to the process for remelt. Railcars and tankers are loaded in the shipping area, which is a covered building

and equipped with sliding gates on both sides of the building. Figure 3.19.12 shows a railcar during loading and Figure 3.19.13 shows a tanker for loading sugar. The weight of the loaded sugar is determined by weighing the vehicle before and after loading.

At the customer's facility, the sugar is unloaded by floor hopper or pneumatically, depending on the type of transport.



Figure 3.19.12 Loading of a railcar with sugar



Figure 3.19.13 Sugar tanker

SAFETY PRECAUTIONS

Safety precautions must be carefully followed in the areas where sugar is dried, packed, moved, stored, and the areas where specialty sugars are produced. These areas are the riskiest places in the factory with regard to **dust explosion** (sudden fire).The concentration of sugar **dust** (particles less than 500 microns), the size of dust particles, and air humidity are the factors affecting the explosion occurrence. The amount of dust in the air must not exceed the minimum explosion dust concentration ($20\,g/m^3$). The size of dust particles is also important (*the smaller the dust particles, the higher is the risk of explosion and the more violent the explosion*). In the past, several major sugar-dust explosions have occurred in sugar factories, causing loss of life and extensive property damage. Investigations proved that almost all began with a small explosion and increased because *fire follows pressure rise*, creating more explosions on its way. The following are few safety precautions:

- **Housekeeping:** The most important precaution is housekeeping. Sugar screening, packing, and the area under the conveyors should be cleaned regularly. Dust should not accumulate on equipment, walls, and tanks.
- **Adequate ventilation:** Adequate ventilation should be provided to control airborne (in the air) dust accumulation.
- **Electrical system:** All electrical devices installed in related equipment should be explosion-proof and confirm with the highest security codes.
- **Relative humidity:** The recycling air to the sugar silo should contain at least 50% RH and should be filtered to remove dust. A constant addition of fresh air is required.
- **Welding and grinding:** Welding, grinding, and drilling should be avoided in dusty areas. When these activities are necessary, the area should be cleaned first, and the entire process should be supervised by the safety manager of the factory.
- **Dust collectors:** Sugar with too low moisture-content (lower than 0.03%) makes dust by abrasion during movement. Therefore, **dust collectors** (scrubbers) should be installed in the sugar dryer, cooler, silo, conveying equipment, and mills for powdered sugar production.

OPERATING PROBLEMS

The station containing drying, screening, packing and storing is a large operation. By solving a problem in one process, you can prevent its adverse effect on other parts of the station. When a problem occurs, evaluate the causes of the problem and attempt to solve it promptly. Described below are the **problems** that can occur during the sugar drying and storing.

Lumpy Sugar after Drying

If the sugar is lumpy after drying, the following problems may exist:

- **High moisture in wet sugar:** If wet sugar leaving the centrifuge has high moisture (more than 1.5%), the dryer cannot dry the sugar sufficiently. In these situations, the washing time at the centrifuge should be decreased.
- **Low dryer capacity:** If the load of sugar is higher than the dryer's norm, the dryer cannot sufficiently dry the sugar.

- **High moisture content in drying air:** If drying air has a high moisture content (more than 60%), the temperature of the drying air should be increased.
- **Low drying airflow**
- **Short resident time**
- **Low drying temperature**

Increased Moisture Content of Stored Sugar in Silo

The following are a few causes of increased moisture content of stored sugar:

- **High temperature of the ventilation air:** If the temperature of the ventilation air entering the silo is higher than 30°C, the equilibrium condition between the sugar crystals and the surrounding air is interrupted, increasing the moisture content of the stored sugar. In addition, the color of the sugar is increased. In these situations, the ventilation air should be cooled to 20 to 25°C.
- **High moisture content in the ventilation air:** To avoid moisture accumulation, the moisture content of the ventilation air should not exceed the limit (60%) in any place in the silo.

High Color Development in Stored Sugar

The usual causes of high color development of stored sugar in the silo follow:

- Sugar has high pH (above 7.3)
- Ventilation air has a high relative humidity
- Temperature-profile across the silo is not uniform
- High temperature in storage area (more than 30°C)
- Sugar has a high moisture-content (more than 0.03%)
- Sugar has a high content of invert sugar (more than 0.02%)

Condensation in the Walls of the Silo

High condensation can occur in silos that do not have heated walls. In such silos, the circulation air to the silo should be dried to prevent condensation.

END-OF-THE-SECTION NOTES

- **Drying, conditioning, and storing bulk sugar in the silo are important issues in sugar production. Sugar factories are not similar with respect to the operating conditions and equipment in their sugar drying and conditioning stations, packinghouses, and sugar warehouses. Variations in sugar quality, the type of dryer-cooler, the silo, climatic conditions, and particularly the sugar-conditioning system existing in different facilities makes it difficult to generalize the subjects related to this station and to optimize its operation uniformly. Therefore, the explanations in this section should be used as a general guide.**
- **Station 19 is the last station for the production of granulated-refined sugar. At this point, the sugar is ready to be shipped to customer in bulk or packed form, or directed to Station 20, where specialty sugars (such as powdered sugar and brown sugar) are produced.**

CHAPTER 3

SECTION 20 PRODUCTION OF SPECIALTY SUGARS

SUBSECTIONS

- Special Crystal-Size Sugars
- Powdered Sugar
- Brown Sugar
- Cube Sugar
- Candy-Crystal Sugar
- Cone Sugar
- Adant Sugar
- Comparison of Cube and Casting Sugars
- Liquid Sugar

ABOUT THIS SECTION **This section** describes **Station 20** (Production of Specialty Sugars) of sugarbeet processing. Station 20 is part of the sugar warehouse where special equipment is installed to produce specialty sugars (see Figure 3.20.1). (Note: A sugar factory may produce one, more, or none of the specialty sugars.)

Specialty sugars are all sugar products except the normal crystal size granulated-refined sugar (GR sugar). This definition includes:

- Special crystal-size sugar
- Powdered sugar
- Brown sugar
- Cube sugar
- Adant sugar
- Candy-crystal sugar
- Cone sugar (loaf sugar)
- Liquid and liquid invert sugar

Beet-Sugar Handbook, by Mosen Asadi
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Figure 3.20.1 Some specialty sugars

In addition, a small demand exists in some countries (particularly in Europe) for the following specialty sugars:

- Organic sugar (produced from organic sugarbeet)
- Unrefined raw cane sugar
- Golden-granulated sugar
- Added-value sugar
- Caramelized sugar
- Swizzle stix sugar
- Muscovado sugar
- Decorative sugar
- Demerara sugar
- Molasses sugar
- Colored sugar
- Instant sugar
- Snowy sugar
- Caster sugar
- Pearl sugar
- Dust sugar
- Jam sugar

Most of us have not heard of all of these sugars because some of them are particular to certain countries. Most specialty sugars are produced from GR sugar to meet specific needs of custom-

ers. The specialty-sugar production lines operate throughout the year by using sugar stored in silos. In addition to higher labor costs compared with GR sugar, the specialty sugars are used less than GR sugar, so their price is relatively high. This section describes the most common specialty sugars.

SPECIAL CRYSTAL-SIZE SUGARS

Special-size sugars are **granulated-refined** (GR) sugar with custom size crystals. Some industrial customers require sugar with extra-fine or extra-coarse crystals for use in their sugar-added products. The special-size (extra-fine and extra-coarse) sugars are sold at premium prices.

Fine sugar dissolves better (the solubility of small crystals is larger than that of the large ones at the same temperature). The baking industry uses extra-fine sugar because it mixes well due to its high surface area and produces dough with a higher rising property, resulting in higher quality baked products (finer texture and fewer crumbs). In some fillings in which the sugar is not fully dissolved, extra-fine sugar is used to avoid grittiness. Chocolate producers also use extra-fine sugar to shorten the milling process because they add sugar to other ingredients before milling.

Special-size sugars are usually produced from fractioning the sugar by passing it through a multiple-screening operation (see the Screening discussion in Station 19). Coarse sugar is produced from the highest purity, lowest color syrup. Usually, part of the initial massecuite is discharged from the pan crystallizer, and crystallization is continued to grow the remaining crystals to the desired size.

As stated in the preceding station, the size of sugar crystals is expressed (in mm or μm) by the **mean aperture** (*MA*), which is the size of the screen opening, on which 50% of the sugar sample stays and 50% passes through. Standard crystal sizes are:

- Extra-fine sugar with an *MA* of about 200 μm or 0.2 mm
- Fine sugar with an *MA* of about 300 μm
- Medium-size sugar with an *MA* of about 500 μm
- Coarse sugar with an *MA* of about 1000 μm
- Extra-coarse sugar with an *MA* above 1000 μm

NOTE

In most countries, fine- and medium-size sugars are the standard, and extra-fine, coarse, and extra-coarse sugars are considered specialty sugars, for which customers pay a premium.

POWDERED SUGAR

Powdered sugar (see Figure 3.20.2) is a mixture of ground GR-sugar and anticaking agent (usually starch), which is added to prevent lumping. Powdered sugars have very fine **amorphous particles** (broken, unorganized, and shapeless molecules) measuring about 50 μm (0.05 mm). Fine particles increase **hydroscopic** (moisture-absorbing) property of the product; therefore, an anticaking agent (in the amount of 3 to 5%) is usually added. Powdered sugar is used in confectionary and chocolate products.

Powdered sugar is produced in three different particle sizes:



Figure 3.20.2 Powdered sugar

- 6X powdered sugar
- 10X powdered sugar
- 12X powdered sugar

In this classification, the higher the number preceding the X, the finer is the particle size. The X refers to the size of the screen through which the powdered sugar was first produced. Specifications for the size of powdered sugars are reported by the amount retained on one screen. (See Chapter 4 for powdered sugar specifications and Chapter 11 for the particle size test.)

Powdered sugar is produced by milling GR sugar in hammer mills. As a rule, *the finer the particles of powdered sugar, the greater is the moisture absorption and the higher is the risk of caking in the bag*. Therefore, finer types need more anticaking agent. To prevent caking and lumping, an anticaking agent is added to sugar during grounding. The typical anticaking agent is **starch**, which is mixed with the sugar at 3 to 5%.

Powdered sugar is produced in a batch operation in a mill in which the sugar is milled for a certain period to produce particles with the desired size. The section for making powdered sugar consists of sugar and starch feeders, mills, and discharge equipment. Sugar, before feeding the mill, is passed through a magnet to remove metal fragments. Mills have different capacities; a medium-size mill can produce 0.5 to 0.8 t of product per hour.

During milling, the sugar temperature increases to about 50°C because of the high mechanical stress between the sugar and milling hammers. To prevent explosions, a current of conditioned air (cool, filtered air with 55 to 65% RH) should be circulated through the mill. In addition, the sugar level in the mill should be low enough to allow the passage of the air current. The air introduced to and flowing out of the mill is filtered to remove dust. To confine a possible explosion in the mill, there are explosion valves at the air input and output, as well as at sugar input and powdered-sugar output.

Like GR sugar, powdered sugar is packed in household and industrial sizes. **Household** (consumer) size powdered sugar is usually packed in 2 lb in the United States and 2 kg polyeth-



Figure 3.20.3 Brown sugar

ylene bags and corrugated for shipping. **Industrial size** powdered sugar is packed in 10 kg (25 lb), 20 kg (50 lb), and 40 kg (100 lb) multi-wall paper bags.

BROWN SUGAR

Brown sugar (see Figure 3.20.3) is a mixture of fine GR sugar and brown cane syrup (food-grade molasses). Brown sugar adds cane-molasses flavor to baking products such as cookies and candies. Because brown sugar has a soft and smooth feel, it is also called **soft sugar**.

Brown sugar is produced in two different ways:

- Crystallization method
- Mixed (painted) method

The **crystallization method** is mainly used by the cane-sugar industry because cane raw sugar contains the special, pleasant flavor required for brown sugar. In this method, a selected syrup is crystallized in the pan to produce a massecuite, and the massecuite is not completely washed during centrifugation to leave some mother-liquor flavor on the crystals.

The **mixed method** is used by both the beet- and cane-sugar industries. In this method, fine sugar with uniform granulation is mixed with cane syrup (molasses) in the proper amount to give the flavor and color desired by the customer. The mixed method is more common because the syrup and sugar are added separately, giving you more control in the formulation. However, the crystallization method is more cost effective.

Brown sugar is of two types:

- Light (golden) brown sugar
- Dark brown sugar

Brown sugar is sold in 1 kg (2 lb in the United States) and 2 kg (4 lb) plastic (poly) bags. For industrial use, brown sugar is packed in 10 kg (25 lb), 20 kg (50 lb), and 40 kg (50 lb), polyethylene-lined, multi-wall, paper bags.

NOTE

Brown sugar should be stored in a controlled environment to avoid lumping. The recommended storage temperature is between 10 and 25°C (50 to 77°F) with 35 to 50% humidity. The product should be used within 45 to 60 days from the date of purchase.

CUBE SUGAR

Cube sugar is a cubical-shaped sugar, produced by pressing moist granulated-refined (GR) sugar in a mold, so cube sugar is also called **molded cube sugar**. A typical cube sugar measures 20 × 20 × 12 mm and weighs about 5 g, but it comes in different sizes and shapes. (Cube sugar in the shape of playing cards is called **bridge cube sugar**.) Cube sugar is mainly for household usage and is not used industrially in sugar-added food products. Cube sugar comes in 0.5 kg (1 lb in the United States) and 1 kg (2 lb) packages (see Figure 3.20.4).

In a typical line for production of cube sugar, GR sugar is moistened in a screw conveyor by adding 2 to 3% water (depending on the desired firmness of the cubes). The moist sugar is fed to a mold, and the mold is heated uniformly, allowing the moisture to escape evenly. As a result, the sugar crystals bind together to produce a hard cube sugar. The most important factors in producing cube sugar are the following:

- Crystal size of sugar
- Amount of water (or steam) added to the sugar before pressing
- Pressure of the press applied to the cube
- Drying time

These factors are important because they determine the firmness, stability, and dissolving rate of the cube when it is used (see the Note below).



Figure 3.20.4 Cube sugar

To improve the binding process between the sugar crystals and to stabilize the cube's form, sugar with a wide distribution size is used in the production of cube sugar. A wide distribution is the term used for sugar with about half its crystals in medium size (MA = 500 micron); the remaining crystals are on the coarse side.

The system for making cube sugar is known as **line** because the process is continuous and systematic. Lines for cube-sugar production can produce 10 to 50 t cube sugar per day. The following are the most common processes for the production of cube sugar:

- Vibro process
- Chambon process
- Elba process

The **vibro process** was developed by the Swedish Sugar Corporation. In this process, a vibrating action presses the moist sugar into molds. A second vibrating action removes the formed cubes from the molds. The cubes are then moved to an oven, which uses radiation to heat the cubes to remove moisture and harden them. The cubes are then cooled and packaged automatically.

The **Chambon process** was invented by French sugar technologists. Its processing line consists of a screw conveyor in which the sugar is moistened with water to 1.6 to 2% moisture content. The screw supplies the moist sugar to a rotating molding unit. During the first half of the rotation, the cubes are pressed between plungers. During the next period, the cubes are moved out of the molds. The cubes move to a vertical dryer and cooler and remain there for about 30 minutes at about 70°C. After cooling, the cubes are placed automatically in cartons in a three-layer arrangement. Ready cartons are marked with a code date and wrapped in plastic foil in groups of 10 to 12 units. A Chambon line can produce up to 50 t cube sugar per day.

The **Elba** processing line operates similarly to Chambon with a rotating molding unit. This line has a dryer heated with infrared energy. The dryer heats the cubes to about 70°C. In the cooling section, the temperature drops to about 40°C.

NOTE

In most countries, people put a cube of sugar into a cup to sweeten their tea, so they want a cube that dissolves quickly. In some countries, however, people put a cube sugar into their mouth where it slowly dissolves while they drink tea, so they want hard cube (see Figure 3.20.5).

CANDY-CRYSTAL SUGAR

Candy-crystal sugar consists of large sugar crystals 5 to 20 mm that are white or brown (see Figure 3.20.5). (Note: Candy crystals differ from rock candy.) The crystals in candy-crystal sugar are separate, but rock candy consists of connected large crystals, usually formed on a string during production (refer to Station 16 for rock-candy production).

Candy-crystal sugar is produced according to the following procedure:

1. High-quality GR sugar is melted in pure water in a heated tank with a stirrer at about 90°C to produce a saturated liquor with 80% *DS*.



Figure 3.20.5 Candy-crystal sugar

2. The liquor is directed to a cooling crystallizer. Here, the temperature of the liquor is lowered to produce crystals and give them time to grow. The slow cooling crystallization continues for 10 to 12 days.
3. After this period, the mother liquor is discharged.
4. The crystals are moved to a crystallizer with a stirrer and saturated pure sugar liquor is added. Here, the crystals grow under slow stirring to the desired size. This stage takes 5 to 6 days.
5. The mother liquor is discharged, and the large crystals are separated from the remaining mother liquor in centrifuge.

The yield of the operation is about 25% of the input sugar. The production of brown candy crystals is the same as that of white candy, except caramel is added during the first stage of operation.

CONE SUGAR

Cone sugar (loaf sugar) is a cone-shaped, molded sugar with a smooth surface that is produced by a pressing or casting process using a special mold (form). Cone sugar (see Figure 3.20.6) is produced in 0.25 kg and 2 kg sizes (and was once produced in 0.25 kg, 2 kg, 5 kg, and 12 kg sizes). Cone sugar is still popular in some countries for its firmness and because it is less hydroscopic (moisture absorber), making it easier to transport. (In other countries, such as the United States, cone sugar has never been popular.)

In Germany, cone sugar is produced by just a few sugar factories and is used traditionally for production of special hard liquor. In some countries in Asia (e.g., Iran) and Africa, cone sugar is still popular because of its hardness and compact form.

Cone-sugar molds were one of the first molds produced by humans. Around AD 600, the Persians invented a cone-shaped clay mold for the production of cone sugar. Cone-sugar molds made of sheet metal are still used in some countries. The consumer cuts the cone sugar first

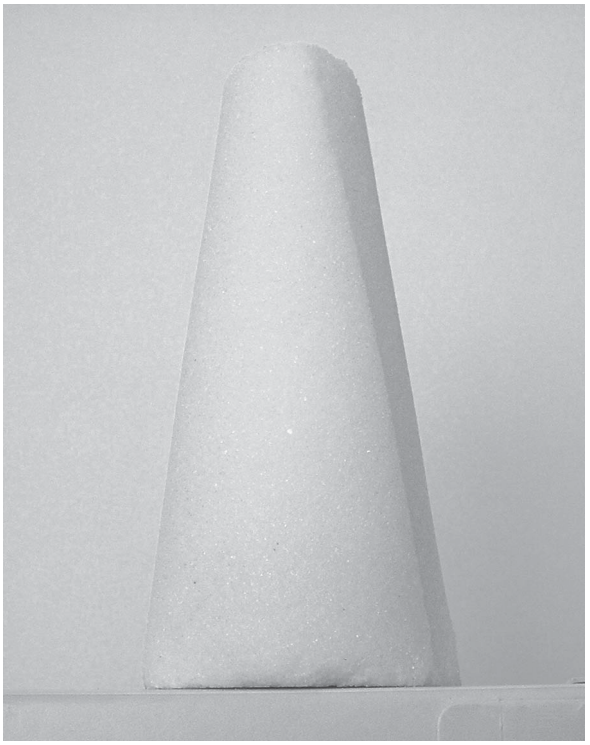


Figure 3.20.6 Cone sugar

into larger pieces with a cast-iron pincer and then into regular cube-sugar size with a little chopper. Cone sugar is produced by one of the following ways:

- Pressing of granulated-refined sugar
- Casting of a pure sugar solution

The **pressing process** is similar to the production of cube sugar. Moist fine sugar is pressed into the molds by a pneumatic action from below. Loaf-sugar **molds** (see Figure 3.20.7) are made of a galvanized metal sheet 1 mm thick. Molds for producing a 2 kg loaves are 275 mm high with 135 mm in diameter at the top, which is the widest part of the mold. The mold has a small dripping hole (a hole 4 mm wide) in the middle of its bottom, so that the syrup around the crystals can dripped out slowly.

During filling, the dripping hole is closed with a plug. The formed loaves are taken from the molds manually and put on moving plates directed to a tunnel dryer. To prevent cracking in the loaves, the drying process is performed slowly over a 24-hour period at 22 to 25°C. The dried cones are placed manually into a wrapping machine, which covers them with a clear plastic wrap. The wrap is folded at the bottom and sealed.

In the **casting process**, the lowest color and highest purity liquor in the sugar-end is used to produce cone sugar. The liquor is crystallized and the massecuite is discharged at about 93% *DS* and a temperature of about 98°C. From this stage onward, the processing is different

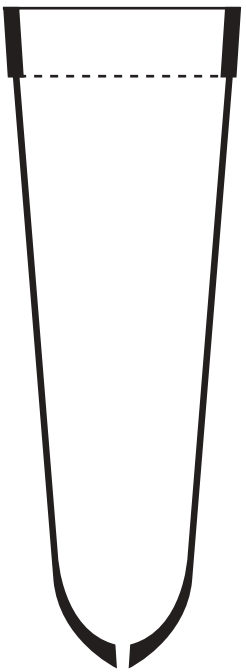


Figure 3.20.7 Cone-sugar mold

compared with traditional sugar-end operations. The production of cone sugar using the casting process is a labor-intensive and high-cost operation, and its production line is not fully automated because of low demand. The operation consists of the following steps:

1. The molds are placed with their tips down on filling trucks and moved to the **filling room**, where they are filled with the massecuite to about 3 cm below their brim. The molds for making 2 kg cone sugar hold about 3 kg massecuite (the density of the massecuite with 93% *DS* is about 1.5 kg/L). Before filling, the mold's dripping hole is closed with a plug.
2. The trucks are moved to the **holding room** for further crystallization of the massecuite by cooling. The temperature of the holding room is kept at 40°C. The holding time is 5 to 8 hours, depending on the ventilation in the holding room.
3. During the cooling period, the massecuite is mixed three times because the crystals move to the bottom and the mother liquor stays on the top. Therefore, the mixing process improves cooling crystallization. During the holding period, the temperature of the massecuite drops from 98 to about 60°C. After the holding period, the massecuite contains around 70% crystals. The remaining content is mother liquor, which is nearly saturated at this temperature.
4. The trucks are moved to the **washing room** to improve the quality (increase the purity and decrease the color) of the mother liquor. The quality of the mother liquor is improved by displacing the low-purity, high-color mother liquor present in the molds with a high-quality liquor, called **washing liquor**. In the washing room, the plugs are taken out of the mold bottom so that the mother liquor can gradually drip from the mold. When the level of the massecuite is lowered sufficiently, the molds are refilled with a filling liquor. The **washing**

liquor is prepared by melting the lowest color GR sugar in hot water to produce a liquor with about 70% *DS*, which is slightly unsaturated (with a supersaturation coefficient, K_{SS}, of 0.98). If the color of the liquor is high, it is decolorized by filtering it through active carbon. The holding time in the washing room is about 12 hours, during which the molds are refilled three to four times with the washing liquor. (Note: During the washing period, neither crystallization nor dissolving of sugar crystals is desired.) Further crystallization increases the crystal content of the massecuite, which can plug the dripping hole of the molds. Dissolving sugar that has already crystallized is not desired because it decreases the firmness of the cones, which can crack during centrifuging. To prevent these difficulties, the temperature of the washing room and washing liquor should be kept at 40°C, and the concentration of the washing liquor should be 70%.

5. The trucks are moved to the **centrifugal station**, and the molds are placed in special centrifuges (described later), each holding 140 molds. The working cycle of the centrifuge is about 12 minutes, including loading and unloading of the forms. Toward the middle of centrifuging, the cones are washed with the washing liquor. The loaves contain 2 to 3% moisture after the centrifugal operation.
6. The loaves are taken from the molds and placed on drying trucks, which move them to a tunnel-type dryer consisting of drying and cooling sections. The temperature is about 55°C in the drying section and 35°C in the cooling section. The drying and cooling time is about 72 hours. The drying-cooling equipment has a capacity of 300 to 600 cones. After the drying and cooling process, the cones contain 0.2 to 0.3% moisture.
7. After drying, the loaves are weighed. If they are too heavy, the bottom of the cone is ground until it is at the desired weight. The powder from grinding is used in the production of powdered sugar.
8. The loaves are then wrapped in normal packing or transparent paper.

The **centrifuges** for spinning molds (see Figure 3.20.8) are bottom-driven by a motor. The drum consists of horizontal nests for holding the molds. Each nest has a hole that matches the

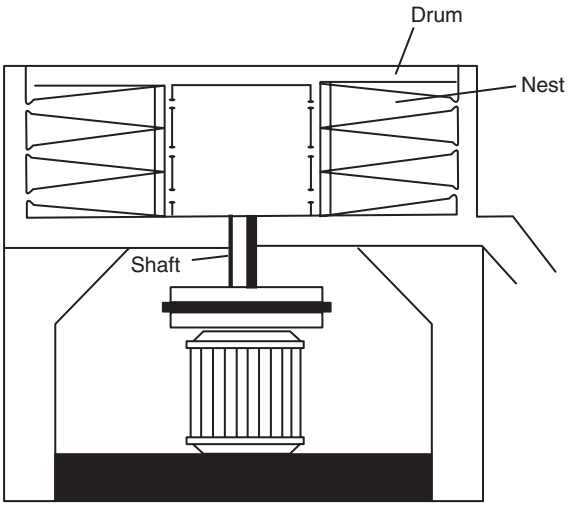


Figure 3.20.8 Cone-sugar centrifuge

dripping hole in the mold for discharging the mother liquor. There are three to four rows of nests, one above the other, and each row contains 10 nests.

The drum of the cone-sugar centrifuge rotates at 500 to 600 RPM to develop **centrifugal force**. The **separation factor** (Z, the ratio of centrifugal force to gravitational force) expresses the separating power of the centrifuge. The Z for a unit of mass is given (see Station 18)

$$Z = \frac{D \cdot n^2}{1800} \tag{3.20.1}$$

EXAMPLE 1

Given:

Drum diameter	2.0 m
Revolution per minute (n)	500

Calculate:
Separation factor (Z)
Operating cycle time

$$Z = \frac{D \cdot n^2}{1800} = \frac{2.0 \times 500^2}{1800} = 278$$

This means that the molds are under a centrifugal force 278 times greater than the gravitational force. Assume that the time of free separating (the time for separating the mother liquor without centrifuging) is 50 hours (3000 minutes). Then, the separating time is 278 times shorter. Therefore, the operating cycle of this centrifuge is

$$\frac{3000}{278} = 11\,\text{min}$$

(Note: The preceding calculations are based on approximate values; in reality the operating cycle will be different depending on the type of centrifuge.)

ADANT SUGAR

Adant sugar (named after its German inventor G. Adant) or **chipped-slab sugar** is molded sugar with a rough surface and is produced by casting and chopping. Usually, four of six faces of cubes of Adant sugar (see Figure 3.20.9) are smooth (due to molding), but the remaining two are rough (due to chipping with a nipper). Adant sugar was popular before the invention of cube sugar. Compared with cone sugar, it is more suitable for chipping to smaller pieces because it is produced in a **slab** (a flat piece) shape about 20 mm (3/4 in.) thick.

Adant sugar is produced from a saturated solution of GR sugar and sugar syrup. The procedure follows:

1. A very high quality massecuite is produced by mixing medium-sized GR sugar with high-quality sugar liquor.
2. The hot massecuite is poured into a special mold (about 36 cm long, divided in 10 sections by dividers 2 cm apart, as shown in Figure 3.20.10) to form a slab about 2 cm thick.



Figure 3.20.9 Adant sugar

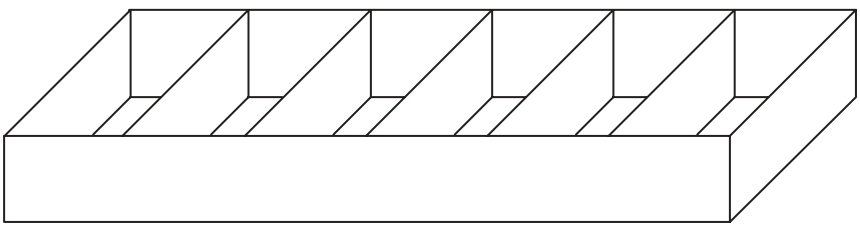


Figure 3.20.10 Mold for Adant sugar

3. The massecuite is given 8 to 12 hours time to solidify at about 40°C.
4. The molds are centrifuged in special centrifuges.
5. The slab bars are taken from the molds and placed on drying trucks, which move them to a tunnel-type dryer consisting of drying and cooling sections.
6. Automatic choppers cut the cooled slabs to the desired cube-sugar size.

COMPARISON OF CUBE AND CASTING SUGARS

It is useful to compare the properties of cube sugar and casting sugars. The advantages of cube sugar over casting sugars are:

- **Ease of operation:** Cube sugar is much easier to produce than casting sugars.
- **Processing costs:** Cube sugar can be produced for as much as one-third the cost of producing casting sugar.
- **Labor costs:** Labor costs for producing cube sugar, which has a fully automated line, are less than those for casting sugar, which is a labor-intensive operation.
- **Labor comfort:** The station for the production of cube sugar has an air-conditioning system and is one of the cleanest places in a sugar factory. The washing room for cone-sugar production has uncomfortable conditions, with temperature of 40°C and high humidity.
- **Desired firmness:** Lines for production of cube sugar can be maintained to produce soft or hard cube sugar.
- **Losses:** The line for the production of cube sugar is highly efficient, but casting production has a high sugar loss.

It is no wonder that soon after the invention of fully automated cube sugar lines, casting-sugar production gradually came to an end in some countries. Countries that still produce casting sugars do so in only small quantities to cover special demands.

LIQUID SUGAR

Liquid sugar is a solution of sugar or invert sugar in water with a definite concentration, mostly for industrial use. Liquid sugar was first produced in 1927 in the United States (McGinnis 1982). Liquid sugar has the following advantages over GR sugar:

- Is easy to transport, handle, and measure
- Can be added directly to food products without melting
- Makes operations of sugar-users, such as the beverage industry, easier

The disadvantages of liquid sugar over GR sugar are as follows:

- About one-third of the transportation cost is for water (liquid sugars are made at about 66% *DS*)
- Tanks for storing liquid-invert products should be acid-proof due to their low pH
- Liquid sugars have a low stability against microbial activities

The sugar industry produces three types of liquid sugar:

- Liquid sucrose
- Liquid invert sugar
- Liquid blend sugar

Liquid Sucrose

Liquid sucrose is a solution of sugar (sucrose) in water with a concentration of 66%. (Note: At 20°C, the maximum solubility of sugar in water is 66.7%; see Table A.2 in the Appendix. So sucrose solution is produced slightly undersaturated to prevent the risk of crystallization during storage or transport.) Liquid sucrose production consists of the following steps:

1. High-quality GR sugar is melted in pure water in a heated tank with a stirrer at about 85°C to produce a solution with 66% *DS*.
2. The solution is filtered or screened through a fine screen.
3. The filtrate is briefly heated to sterilize.
4. The product is cooled and stored in a storage tank.

Some factories prepare the solution slightly higher than 66% and then evaporate the syrup to the desired concentration of 66%. This approach is better at sanitizing the syrup against microbes.

Liquid Invert Sugar

Liquid invert sugar is a solution of sugar and invert sugar in water. Liquid invert is commonly used as a sweetener. Invert sugar is a mixture of equal parts of glucose (dextrose) and fructose (levulose) produced from sucrose inversion. At the same concentration, liquid invert is slightly sweeter than liquid sucrose and gives food products a pleasant flavor. Liquid invert is used in food products such as baking products to hold moisture. Liquid invert sugar increases the shelf life of food products.

(More about the inversion of sugar [sucrose] in acid solutions is given in Section 4 of Chapter 1.) For now, remember that the sucrose inversion belong to the **hydrolysis** (the reaction of a compound with water) group of reactions and proceeds according to the following equation:

$$\underset{\text{Sucrose}}{C_{12}H_{22}O_{11}} + \underset{\text{Water}}{H_2O} \rightarrow \underset{\text{Glucose}}{C_6H_{12}O_6} + \underset{\text{Fructose}}{C_6H_{12}O_6}$$

Sucrose	Water	Glucose	Fructose
342	18	180	180

As seen from this equation, one molecule of water is required for the inversion process, which increases the weight of product by 5% ($18/342 \times 100 = 5\%$).

Liquid invert products are different in invert sugar content (from 10 to 90%). The most common product is liquid invert with 50% invert and 50% sucrose (on solid base), which equates to 38.5% invert, 38.5% sugar, and 18% water.

The sugar industry produces liquid-invert products in two ways:

- Hot acid method
- Strong-acid-cation (**SAC**) resin method

The second method has little application, so only the first one is described here. Sugar hydrolyzes (reacts with water) easily in acid solutions to form invert sugar. The **inversion rate** (the change in invert-sugar concentration per unit time) depends on the following factors:

- Concentration
- Temperature
- pH

To decrease the pH of the solution, you add acid (usually HCl). However, the acid plays a role of a **catalyst** (a substance that speeds up a reaction without being consumed), so the reaction can be considered the reaction of sugar with water rather than sugar with acid.

Following is the procedure for producing 50% liquid invert using the batch method:

1. In the inverting tank equipped with a stirrer, high-quality sugar is mixed with pure water in a ratio of three to one. This ratio prepares a 75% liquor.
2. The tank is heated to about 70°C while the liquor is stirred.
3. Sufficient concentrated hydrochloric acid (HCl) is added until the pH drops to 4.5.
4. Additional small amounts of acid are added (about 20 mL per each 100 kg of sugar processed) and timing starts.
5. After 30 minutes, a sample is taken and tested for invert sugar content. Until the desired inversion level is reached, a sample is tested.
6. Sufficient 15% NaOH solution is added to adjust the pH to 5.5. Usually, about 75 mL of 15% NaOH are required for each 100 kg of sugar processed.
7. The concentration of the product is adjusted to 66%.

Liquid Blend Sugar

Liquid blend sugars are liquids that contain other carbohydrates in addition to sugar and invert sugar. Depending on the **dextrose equivalent** (DE, the relative amount of glucose, fructose, and other reducing sugars in blended products) value, the viscosity of the blended products is high. The sugar industry produces different types of liquid blends according to the customers' requirements.

Transportation of Liquid Sugar

Liquid-sugar products have a low **pH**, so they are stored and transported in stainless steel tanks. The products are transported to customers in tank trucks and railcars. Tank trucks can handle up to 25 t of products (see Figure 3.20.11). At 70% concentration and 1.35 density, this equals 18.5 m^3 or 5000 gallons of liquid sugar. Tanks are bottom-loaded and bottom-unloaded using a gas-driven pump.

The tank and parts in which the liquid sugar flows (e.g., pump and pipes) should be cleaned (with alkaline cleaning agents followed by rinsing) after each unloading if different products are carried. If the same products are carried, cleaning should be performed every day or two.

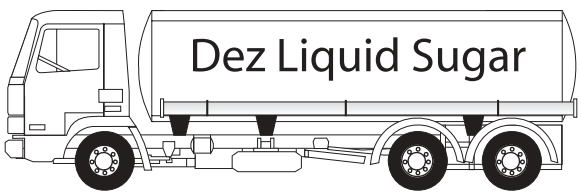


Figure 3.20.11 Liquid-sugar tank truck

END-OF-THE-CHAPTER NOTES

- **Chapter 3 consisted of 20 sections, each corresponding to a station of sugarbeet processing. The stations were explained in a systematic order, from the point when beets enter the factory to the point when sugar goes in the silo or bag and specialty sugars are produced. In the next chapter, you will learn how sugar-factory products (sugar and specialty sugars) and by-products (molasses, pulp, and carbonation lime residue) are quality-controlled to keep their integrities.**

CHAPTER 4

QUALITY CONTROL

SECTIONS

- Standards and the HACCP Program
- Quality Control of Sugar
- Quality Control of Powdered Sugar
- Quality Control of Brown Sugar
- Quality Control of By-products
- Quality Control of By-products from Molasses-Desugaring Process
- Nonconformance Procedures and Red Tagging
- Weight Control

ABOUT THE CHAPTER **Chapter 4** gives **specifications** (standards) for the products (sugar and specialty sugars), by-products (pulp, carbonation-lime residue, and molasses), packaging, and warehousing of the beet-sugar factory. The specifications of betaine and raffinate—by-products of the **MDC process** (molasses desugaring by chromatographic process)—are also explained.

The sugar industry, as a food-product producer, is required to operate the sugar factories under two programs to ensure the food products are of high quality and safe:

- **CGMP** (Current Good Manufacturing Practices)
- **HACCP** (Hazard Analysis Critical Control Points)

Both programs are explained in this chapter. Initial steps to establish an HACCP program and its preventive measures such as **nonconformanc**e (out-of-spec) procedure, red tagging, weight control in a sugar facility are also described.

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The 7 HACCP Steps



- Evaluate the facility for the HACCP program
- Identify the critical control points (CCP) of the facility
- Establish critical limits for preventive measures of each CCP
- Establish procedures for control and maintaining each identified CCP
- Establish corrective actions for occasions that a deviation occurs in any of CCP
- Establish effective record-keeping procedures for completion of the HACCP program
- Establish verification procedures to assure that the HACCP program works properly

The quality of the food we consume is important. **Sugar** (sucrose) is a basic carbohydrate that each of us consumes every day, either directly or indirectly in sugar-added food products. Sugar customers take quality seriously and so must the producers. The sugar producers have an obligation to follow two programs to ensure their products are of high quality and safe:

- **CGMP** (Current Good Manufacturing Practices): Determines the current sanitary standards for manufacturing, storing, and packing to ensure the food products are safe.
- **HACCP** (Hazard Analysis Critical Control Points): Determines critical control points (CCP) of the facility, preventive measures of each CCP, nonconformance (out-of-specs) procedures for products, and hazards (risks) related to safety and quality of products in a food facility.

STANDARDS AND THE HACCP PROGRAM

Since the 1990s, the world sugar industry has been trying to incorporate the HACCP (Hazard Analysis Critical Control Points) program and the **ISO** (International Standards Organization) 9001 regulations into the day-to-day operations of their facilities to ensure products safety and customer expectations. HACCP is a quality and safety program designed for the food and pharmaceutical industries; ISO offers general standards for all industries. The three common standards incorporated in the HACCP and ISO programs used by the sugar industry are as follows:

- **Government standards:** Established by government agencies and generally mandatory. For example, the Food and Drug Administration (FDA) requires that the amount of sulfur dioxide in food products not exceed 20 ppm.

- **Customer standards:** Established by customers and followed by the producer to meet the customer's requirement.
- **Industry standards:** Established by the industry (in our case, sugar industry) to meet government and customer standards.

The most important quality-control standards for the sugar industry are the National Soft Drink Association (**NSDA**), United States Pharmacopia (**USP**), and Canners standards. NSDA standards (known as **Bottlers standards**) for sugar to produce nonalcoholic drinks (soft drinks) were first offered in 1953 and revised in 1975 (Griffiths et al. 1997). Today, these standards are used by most sugar producers throughout the world.

HACCP is a broad program for definition, control, and documentation of critical points and their hazards (risks) related to safety and quality of products and process control in food facilities. The three important initial steps to establish an HACCP program in a sugar facility follow:

- **HACCP team:** First, you should create an HACCP team. The person in charge of quality control of the company usually coordinates the HACCP team. The team might consist of the chief chemist, the packing-and-warehousing (P&W) department manager, and the safety manager.
- **Training:** The coordinator of the HACCP team must be trained by an institute certified by the International HACCP Alliance (see the following Note). If possible, all members of the team should receive training, so they know good practices from bad ones.
- **HACCP zone:** The sugar storage (silo) can be the best starting point for HACCP implementation. From that point on, all operations, persons, and equipment are in the HACCP zone in which critical points must be met. For example, all employees working in the HACCP zone and visitors entering this zone must comply with the HACCP dress code (hair nets and beard guards must be worn and jewelry must be removed).

Following the determination of the HACCP zone and preparation of a flow diagram of the HACCP zone, the following is implemented:

- Documentation of critical points (DCP)
- Labeling worksheet form for products
- Material safety data sheets (MSDS)
- Metal detector check procedure
- Customer complaint procedure
- Sanitation program procedure
- Magnet inspection procedure
- Chemical control procedure
- Weight control procedure
- Pest control procedure
- Loading procedure
- Shipping procedure

Documentation of critical points (DCP) and HACCP procedures are important because customers usually audit the facility by touring it to assess current performance on the quality of products and by-products and food-safety programs. Figure 4.1 shows an example of an HACCP master program. Note: The following HACCP master program is not complete, and it should

Processing step	Potential hazard		Preventative measures	Critical control point
Sugar silo	Biological	Sugar does not support bacterial growth because of its low moisture	Microbiological testing is performed on weekly composite samples	No
	Chemical	No bulk chemicals used	Cleaning products and other chemicals are not permitted in main storage area from the silo without authorization	No
	Physical	Metal contamination	Magnets and screens are located downstream from silo	No
Sugar to packing station	Physical	Metal contamination	Magnets are located above the Sweeco screens	Yes
Retail line	Physical	Metal contamination	Metal detectors are checked every 2 hours	Yes
Industrial line	Physical	Metal contamination	Metal detectors are checked every 2 hours	Yes
Note: Table is not finished.				

Figure 4.1 HACCP master program

be completed individually based on the factory's production lines (e.g., powdered, brown, and cube-sugar lines).

The HACCP team coordinator's job includes the following tasks:

- Inspecting the HACCP zone on a regular basis to identify potential problems
- Informing the person in charge to take care of the problems
- Training employees who work in the HACCP zone
- Controlling chemicals used in the HACCP zone
- Supervising the cleaning of the HACCP zone
- Responding to all customers' complaints
- Supervising a product's recall program
- Supervising the pest control program
- Supervising the sanitation program

The **customer-complaint program** refers to a procedure the company follows when a complaint is made. The following summarizes the customer-complaint procedure:

- The complaint is directed to the marketing (sales) department.
- The complaint is investigated to track the affected product by lot number.
- The complaint is directed to the appropriate department for corrective actions.
- The complaint is investigated to determine whether it is a quality or food-safety issue.
- The sales coordinator responds to the customer to report the corrective actions under taken.

NOTES

- **In most countries, private companies offer introductory and advanced HACCP courses, certified by the International HACCP Alliance. These courses also contain references to ISO 9001 applicable to feed quality. Most of these companies also offer on-site training and audit programs.**
- **Sugar factories usually contract with an outside pest company to maintain pest control.**

Examples of a material safety data sheet (**MSDS**) and labeling worksheet for sugar are given in Figures 4.2 and 4.3.

The following sections describe residve quality standards for sugar, specialty sugars, and the by-products (pulp, carbonation-lime residue, and molasses) of the beet-sugar factory. (Note: The quality standards given are typical and approximate.)

QUALITY CONTROL OF SUGAR

Granulated-refined sugar (GR sugar) is pure sucrose ($C_{12}H_{22}O_{11}$) with a number of impurities in trace amounts. Color, ash, moisture, sediment, and specks are the most common impurities. The standards for GR sugar are listed in Figure 4.4. Note that these are a combination of data from **NSDA**, **USP**, and sugar-industry standards.

Bulk density is the mass per volume of a *loose material*. The bulk density of sugar depends on its crystals size (*the finer the crystals, the larger is the bulk density of sugar*). The following table gives the approximate **bulk density** of fine, medium, and coarse sugars.

Fine sugar	880 kg/m^3 (55 lb/ft^3)
Medium sugar	860 kg/m^3 (54 lb/ft^3)
Coarse sugar	840 kg/m^3 (52 lb/ft^3)

Color is the first characteristic of sugar that attracts our attention. The main function of sugar is to give **sweetness**, not color. The yellow color in sugar is due to the presence of impurities, such as caramels and the decomposition products of invert sugar destruction.

Because gloss and crystal size affect reflectance, it is difficult to measure the color of sugar in its solid state. Instead, color is measured in a 50% sugar solution against distilled water as a reference, with a spectrophotometer. To remove the impurities, the sugar solution is filtered prior to color measurement (refer to Chapter 11 for the analytical procedure).

The following factors affect the amount of color in sugar:

- Quality of the beets
- Juice purification process
- Juice evaporation process
- Amount of coloring substances

A **high-color** problem in sugar can be solved by careful carbonation, by increasing the addition of sulfur dioxide, by controlling the pH, and, to a lesser extent, by additional wash water during centrifuging.

Turbidity (cloudiness) in sugar is generally due to very small undissolved-particles of insoluble limesalts (hardness) or filter-aid leakage. The sugar solution must not be turbid. You can eliminate turbidity by reducing limesalts in the second carbonation and using proper filtration.

Ash consists of soluble and insoluble salts of organic and inorganic compounds (mostly oxides and sulfates of K, Na, Ca, and Mg cations). Some ash comes with the beets and is not eliminated during sugarbeet processing, some ash is added during the process, and some ash is due to processing equipment erosion.

The ash content of sugar must not exceed 0.015%. You can reduce ash content by maintaining a proper filtration, sufficient washing during centrifuging, and proper handling of the sugar during drying and screening. Processing quality beets is also important because a large part of the ash comes from the soluble components in the beet that are not removed during the purification process.

The **moisture** content of sugar must not exceed 0.03%. Sugar with no more than this amount of moisture and temperature of 20 to 25°C is safe for storage, transport, or packing, unless it is exposed to extreme temperature and humidity changes. Sugar with a high moisture content becomes lumpy during transport or storage. Proper conditioning after the dryer generally solves the clumping problem due to high moisture content. The control of moisture content of sugar is an important part of laboratory jobs. The standard procedure for moisture content of sugar is oven-drying method (see Chapter 11, the Quality-Control section, for information on testing moisture in sugar).

Sediment refers to water-insoluble and suspended substances in sugar, such as hardness deposits, rust particles, and filter aid. The amount of sediment is determined by using black and white filter discs (filter papers) and comparing the discs with standard sediment discs (refer to Chapter 11, the Quality-Control section, for information on testing sediment in sugar).

Specks refer to materials in sugar that can be seen without a microscope. Caramelized sugar, which appears as a piece of brown color, is an example of a speck. The specification for specks is 3 in 100 g sugar**.** Any speck test in excess of 3 requires immediate correction. Specks are determined by visual inspection (refer to Chapter 11, the Quality-Control section, for information on testing specks in sugar).

Floc refers to the cloudy and turbid precipitate that forms in some sugar-sweetened carbonated soft drinks after standing for several days. In the beverage industry, this situation is called **acid beverage floc** (ABF). ABF is mainly due to the presence of **saponin**, which belongs to the **colloidal group** (colloids are high molecular substances). Saponin comes from the sugarbeet skin, passes through all the processes, and ends up in sugar. If sugar contains more than 2 ppm saponin, it produces visible flocs after standing in an acidified solution for 1 to 8 days (refer to Chapter 11, the Quality-Control section, for information on testing floc in sugar). Sugar with high floc content is not suitable for use in carbonated-beverage production because it forms haze or snowflakes. (Note: Saponin is also largely responsible for foaming in in-process products.)

Saponin is more soluble at a higher pH, so it can be removed during carbonation if sufficiently high alkalinities are maintained. Activated carbon, which is still used in many cane sugar factories for juice decolorization, helps remove the last trace of saponin.

Sulfur dioxide (SO_2) is the only impurity in sugar that enters to the process during operations. It is usually added to thin juice to inhibit the **Maillard** (Browning) reaction, which forms coloring compounds at high temperatures. The Maillard reaction occurs between invert sugar and amino acids during evaporation and crystallization. SO_2 use should be always controlled to ensure that only the minimum amount necessary is added (see the following Notes).

NOTES

- **The FDA (Food and Drug Administration) requires that the amount of sulfur dioxide in food products, including sugar, not exceed 20 ppm. The Codex Alimentarius Standard permits a maximum level of 15 ppm. Sugar should be tested routinely for sulfur dioxide content in the laboratory to adjust the amount of sulfur dioxide used during operation.**
- **The amounts of other trace impurities (e.g., raffinose) in sugar are important. For example during crystallization, a high amount of raffinose ($C_{18}H_{32}O_{16}$, a trisaccharide) alters the crystal shape of sucrose. The raffinose content of sugar produced from damaged beets is usually high.**

Microorganisms are introduced to the sugar by the beets or the process. We are concerned with the presence of two types of bacteria in sugar: mesophilic (with optimum growth between 25 to 40°C) and thermophilic (with optimum growth between 50 to 55°C). Mesophilic bacteria are usually destroyed by temperatures in excess of 85°C. Thermophilic bacteria are capable of encapsulating themselves, and they can often survive boiling temperatures for several hours. Thermophilic organisms are best removed by proper filtration.

Microorganisms are classified as follows:

- Aerobic (need oxygen to grow)
- Anaerobic (do not need oxygen to grow)
- Facultative (can grow with or without oxygen)

To grow, each microbe has an **fermentation process** (the sugar-destroying actions of microbes that produce alcohol at optimum pH). Mesophilic bacteria are the main type of microbes in diffusion. They are mostly aerobic and produce alcohol in the **fermentation process** (the sugar-destroying actions of microbes that produce alcohol or lactic acid).

Crystal Size Distribution

The crystal size distribution (CSD) test with multiple screens is usually performed to determine the average size of sugar crystals and their variation. The production of sugar with minimum variation in crystal size is desired. The sugar industry uses the **coefficient of variation** (*CV*) to mathematically express the size uniformity of sugar crystals. *CV* is a way of describing the **precision** of the result of a set of measurements. Low *CV* value characterizes a better uniformity while a high *CV* value is an indication of a wide and poor distribution size. A *CV* of less than 35% is desirable for sugar crystals.

The **median aperture** (*MA*) is the size of the screen opening through which 50% (one-half) of the sample crystals fall and 50% retain. The *MA* test is based on the cumulative percentages retained on a set of sieves. Results are plotted in graphic paper against the sieve aperture, giving a straight line. The *MA* and *CV* are then determined from the graph (see Chapter 11, the Quality-Control section, for a detailed explanation).

In general, coarse crystals are easier to centrifuge and create a better yield. Very fine crystals pass easily through the centrifuge screen and become part of the mother liquor, which has to be crystallized again, reducing the recovery. The solubility of small crystals is larger than that of large ones at the same temperature, making fine sugar more demanding for industrial use. Sugar factories try to produce **medium-size sugar**, which is 0.35 to 0.40 mm (350 to 400 microns or simply 350 to 400 *MA*) in size.

Material Safety Data Sheet for Sugar

Product	Granulated-refined sugar
Manufacturer	Dez Sugar Company
Chemical components	Sucrose (99.95%)
Chemical formula	$C_{12}H_{22}O_{11}$

Hazardous Ingredients

None

Physical and Chemical Data

Description	Crystals
pH	Neutral in solution
Solubility	Soluble in water (200 g/100 g water at 20°C)
Density	850–900 kg/m^3 (53–56 lb/ft^3)

Fire and Explosion Hazards and Precautions

High concentrations of sugar dust under low humidity may be explosive. Provide adequate ventilation to control dust accumulation. Do not permit smoking or sparks in areas, where sugar dust accumulation may occur.

Reactivity Data

Stability	Stable at normal temperature
Melting point	185°C

Health Hazard Data

Symptoms	NA
Emergency treatment	NA
Carcinogenic	Not carcinogenic

Handling and Control Measures

Storage	Store at 10–25°C (50–77°F) and low humidity (below 70%)
Spill and cleanup	Flush with water
Waste disposal	Observe municipal, state, and federal regulations
Ignition sources	Nonflammable
Housekeeping	Minimize accumulation on floors and equipment
Angle of slope	35–90° (in bulk form)

Figure 4.2 MSDS for sugar

Labeling Worksheet for Sugar

Product	Granulated-refined sugar
	Nutrition Facts
Serving size	One level teaspoon (4 g)
Amount per serving	
	Amount Per Serving
Calories	15
Calories from fat	0
Total fat	0
Saturated fat	0
Cholesterol	0
Sodium	0
Carbohydrates	4 g
Sugars	4 g
Dietary fiber	0
Protein	0

Figure 4.3 Labeling worksheet for sugar

Quality Standards for Sugar

Appearance	White crystals, free of visible foreign materials, dry, and free flowing
Taste	Sweet
Odor	Nonobjectionable in 10% sugar solution in a closed glass container
pH	Neutral or slightly acidic
Sucrose	99.9% minimum
Color	35 IU in 50% solution
Turbidity	20 IU in 50% solution
Ash	0.015% maximum
Moisture	0.03% maximum
Sediment	Maximum 2 ppm on black filter paper and 2 ppm on white filter paper
Specks	3 pieces in 100 g sugar maximum
Floc	2 ppm maximum
Sulfur dioxide	15 ppm maximum

Microbiological Standards

Mesophilic bacteria	Not more than 200/10 g
Yeasts	Not more than 10/10 g
Molds	Not more than 10/10 g

Bulk density	850–900 kg/m^3 (53–56 lb/ft^3)

Figure 4.4 Quality standards for granulated-refined sugar (approximate)

QUALITY CONTROL OF POWDERED SUGAR

Powdered sugar is a mixture of ground granulated-refined (GR) sugar and an anticaking agent (usually starch), which is added to prevent lumping. Three types of powdered sugar are produced: 6X powdered sugar contains large particles, 10X powdered sugar contains medium particles, and 12X has the finest particles. *(The higher the number preceding the X, the finer is the particle size.)* The quality standards for powdered sugars are given in Figure 4.5:

QUALITY CONTROL OF BROWN SUGAR

Brown sugar is a mixture of fine granulated-refined sugar and brown cane syrup (food-grade molasses). **Light** (golden) and **dark brown** sugars are two types of brown sugar. The quality standards for light and dark brown sugars are given in Figure 4.6.

QUALITY CONTROL OF BY-PRODUCTS

The **by-products** of the beet-sugar factory are

- Pulp
- Carbonation-lime residue
- Molasses

Beet pulp is a sugar-depleted and highly fibrous material that is produced after sugar is extracted from sugarbeet. Pulp is a valuable cattle feed and supplies carbohydrates, proteins, and minerals. Pulp (in dry, pelleted, or ensiled form) is used as a high-energy, low-protein supplementary food for **ruminants** (animals, e.g., cows, that bring the food back from their stomach into their mouths for rechewing). In terms of food value, pulp ranks between hay and oats. Its digestibility is 1.5 times higher than hay. The protein digestibility of the pulp is about 75%.

Quality Standards for Powdered Sugar

	6X	*10X*	*12X*
Sucrose (%)	96.0–97.0	96.0–97.0	95.0–96.0
Starch (%)	3.0–4.0	3.0–4.0	4.0–5.0
Ash (%)	0.02	0.02	0.02
Moisture (%)	0.2–0.4	0.2–0.4	0.2–0.6
Particle size			
% on 70 U.S. screen	0.3	0.05	0.01
% through 200 U.S. screen	93.0–95.0	97.0–98.0	99.0 or more
% through 325 U.S. screen			98.0 or more
Bulk density	600–700 kg/m^3 (37–44 lb/ft^3)		

Figure 4.5 Quality standards for powdered sugars (approximate)

Beet-sugar factories sell pulp in various forms:

- Pressed pulp
- Pressed-molassed pulp
- Dry pulp
- Dry-molassed pulp
- Pelleted pulp

The quality standards for dry pulp and pelleted pulp are given in Figure 4.7.

Carbonation-lime residue (CLR, limecake) is another byproduct of the beet-sugar factory. Lime residue is the residual calcium carbonate precipitate left after the juice purification process, followed by pressing process. It is a cake-like substance with about 70% dry substance

Quality Standards for Brown Sugar

	Light Brown	*Dark Brown*
Taste	Mild molasses flavor	Rich molasses flavor
Sucrose	95.0% minimum	95.0% minimum
Invert sugar	1.0–2.0%	1.0–2.0%
Ash	0.3–0.5%	0.3–0.5%
Moisture	1.0–2.0%	1.0–2.0%
Total solids	97.0–99.0%	97.0–99.0%
pH	5.0–6.5%	5.0–6.5%
Color	Golden brown (2500–4000 IU)	Dark brown (8500–11000 IU)
Bulk density	800–880 kg/m^3 (50–55 lb/ft^3)	800–880 kg/m^3 (50–55 lb/ft^3)

Figure 4.6 Quality standards for brown sugars (approximate)

Quality Standards for Dry Pulp

Pectin	46.0%
Fiber	22.0%
Sucrose	6.0%
Protein	7.0%
Ash	5.0%
Minerals	2.5%
Fat	0.5%
Moisture	11.0%
Net energy for gain (NEG)	1140 kcal/kg
Net energy for lactation (NEL)	1790 kcal/kg
Bulk density	200–240 kg/m^3 (12–15 lb/ft^3)
Angle of slope	25–90°

Figure 4.7 Quality standards for dry pulp (approximate)

Quality Standards for Dry-Molassed Pulp	
Pectin	42.0%
Fiber	20.0%
Sucrose	10.0%
Protein	7.0%
Ash	6.0%
Minerals	3.5%
Fat	0.5%
Moisture	11.0%
Bulk density	200–220 kg/m^3 (12–14 lb/ft^3)

Figure 4.8 Quality standards for dry-molassed pulp (approximate)

Quality Standards for Pelleted Pulp	
Pectin	46.0%
Fiber	22.0%
Sucrose	6.0%
Protein	7.0%
Ash	5.0%
Minerals	2.5%
Fat	0.5%
Moisture	11.0%
Bulk density	640–720 kg/m^3 (40–45 lb/ft^3)

Figure 4.9 Quality standards for pelleted pulp (approximate)

(almost the same as soil). It consists of about 80% $CaCO_3$ in its dry substance. about 10% inorganic and insoluble organic compounds, and trace elements, such as selenium, zinc, and molybdenum. CLR can be used as an agricultural soil enhancer and pH adjuster that not only limes (increases pH) the soil but also gives part of its nutritional value back to the soil. It is freed of plant diseases (e.g., nematodes) because of heat during sugarbeet processing.

Carbonation-lime residue has already proved its value in growing several crops including sugarbeets. It is less expensive than other lime suppliers. Besides calcium, it contains other minerals that give extra value to the soil. The lime requirement to control soil acidity depends on the pH of soil. A typical requirement is about 5 t per hectare (2 short t per acre). Quality standards for carbonation-lime residue are given in Figure 4.10.

Molasses is another by-product of the beet-sugar factory. Molasses is a sugar- and non-sugarthick solution that contains about 50% sucrose. Beet molasses must not contain less than 48% sugars (refers to mainly sucrose and invert sugar) and 79.5% *DS* when sold. It usually contains about 50% sugar and 80% *DS*. Because molasses has a high sugar-content and other valuable compounds, it is a valuable raw material in animal feed industry yeast, citric acid, alcohol, and the pharmaceutical industry. Quality standards for molasses are given in Figure 4.11.

Quality Standards for Carbonation Lime

Calcium oxide	45.0%
Carbon dioxide	40.0%
Silica	3.0%
Aluminum oxide	2.5%
Magnesium oxide	1.5%
Ferrous oxide	0.5%
Sodium oxide	0.2%
Potassium oxide	0.1%
Moisture	25.0%
PH	7.5
Neutralizing value	89.0%
$CaCO_3$ Equivalent	460 kg/m^3 (1325 lb/ft^3)
Bulk density	1100 kg/m^3 (69 lb/ft^3)

Figure 4.10 Quality standards for carbonation lime (approximate)

Quality Standards for Nonfood-Grade Molasses

Sucrose	46.0–52.0%
Ash	10.0–12.0%
Protein	8.0–10.0%
Betaine	4.0–6.0%
Water	18.0–20.0%
pH	7.0–7.5
Density (80% *DS*)	1400 kg/m^3 (87 lb/ft^3, or 12 lb/ga)

Figure 4.11 Quality standards for nonfood-grade molasses (approximate)

QUALITY CONTROL OF BY-PRODUCTS FROM MOLASSES-DESUGARING PROCESS

Beet-sugar factories equipped with molasses-desugaring by chromatographic process (see Chapter 8) produce two more by-products (besides pulp, carbonation lime, and molasses):

- Raffinate (also known as CMS)
- Betaine

Raffinate (also called CMS for concentrated molasses solids) contains mainly minerals and low amounts of sucrose and is usually sold to the animal feed industry. Raffinate is sold to customers as 60% dry substance. Quality standards for raffinate are given in Figure 4.12.

Betaine is one of the nitrogenous compounds present in sugarbeet that ends up in molasses. It makes up about 5% of the *DS* in molasses and is one of the most valuable components of sugarbeet. In the molasses desugaring process, a considerable amount of high-purity betaine

Quality Standards for Raffinate

	% on solids
Dry substance	60.0
Sucrose	22.0
Ash	30.0
Protein	21.0
Raffinose	5.0
Nitrogen compounds	3.5
Amino acids	0.5
Others	18.0
Density	1300 kg/m^3 (81 lb/ft^3, or 11 lb/ga)

Figure 4.12 Quality standards for raffinate (approximate)

Quality Standards for Betaine

	% on solids
Dry substance	55.0
Sucrose	2.0
Betaine	50.0
Amino acids	10.0
Organic compounds	10.0
Ash	6.0
Raffinose	1.0
Invert sugar	1.0
Others	20.0
Density	1250 kg/m^3 (78 lb/ft^3, 10.4 lb/ga)

Figure 4.13 Quality standards for betaine (approximate)

can be recovered and sold as a **liquid feed supplement** primarily to the animal feed industry. Betaine also used in the pharmaceutical, cosmetic, and fermentation industries. Quality standards for betaine are given in Figure 4.13.

NONCONFORMANCE PROCEDURES AND RED TAGGING

Nonconformance (out-of-spec) **procedures** are actions that operators follow when the quality of a product does not comply with the specifications. (This type of product is known as an **out-of-spec product**.) Each factory should have written procedures for typical problems that may occur during operations. For example, when a high level of specks (eye-distinguishable materials) are found in the sugar, four reactions must be initiated:

- Separate the unacceptable portion of the product
- Inform the supervisor in charge of the process
- Identify the source of the problem
- Correct the problem immediately

Following is the procedure for responding to high specks in sugar:

1. Sample the standard-liquor tank
2. Filter the sample in the lab and proceed further if specks are found.
3. Sample each standard-liquor filter and take them to the laboratory.
4. Test the samples to find which filter is the source of the problem.
5. Notify management to remove the filter from service for cleaning and change the filter cloth.

During the period when the problem arises until it is solved, all sugar produced that does not meet the standards must be separated (e.g., putting them on pallets) for remelt. Any out-of-spec sugar that has been packaged, the packages must be **red-tagged** (as described next).

Out-of-spec products must be **red-tagged** and separated from the operation line. A product is out-of-spec for any of the following reasons:

- Does not meet product standards
- Does not meet weight control standards
- Does not have correct coding or labeling tags
- Is not correctly palletized, sealed, or packaged

NOTES

- **Out-of-weight (over or under the specified weight) products cause problems for customers, who require exact amounts of ingredients for their recipes.**
- **Red tagging is usually performed by affixing a red label reading: Do Not Ship. A red-tag log must be filled for every pallet of failed product. The red-tag label and a copy of the log must be applied to both of the main two-way entry sides (the side that faces the lift-truck operator when the pallet is picked up and the opposite side) of the pallet. Another copy of the log is filed in the QC logbook.**
- **In all cases, a supervisor must be informed immediately if a product must be red-tagged.**

WEIGHT CONTROL

To control the weight of products, usually one out of every 5 to 10 bags is weighed to check the accuracy of the production line and make adjustments if necessary. If a package found to be under its stated net weight, the next 10 packages are tested. If the average of the 10 weights is below the stated weight, everything produced since the last weight-check is **red tagged**. Figure 4.14 shows the acceptable bag weights for 10 kg sugar bags.

(Note: Example 38 given in Chapter 12 is on weight control and shows how to scientifically decide whether a weight meets the specifications.)

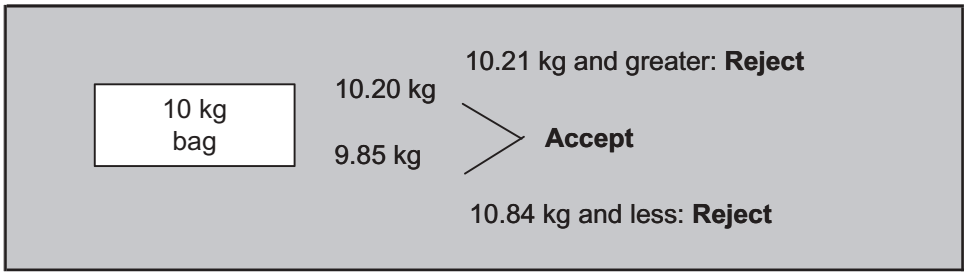


Figure 4.14 Acceptable bag weights

END-OF-THE-CHAPTER NOTES

- In Chapter 3, we learned about sugarbeet processing in a conventional sugar factory, whose product is sugar and by-products are pulp, carbonation lime, and molasses. In Chapter 4, we paid attention to quality control of product and by-products of a such factory. In addition, Chapter 4 made us familiar with the quality control of two more by-products (raffinate and betaine) of the sugar factories equipped with molasses desugaring by chromatographic process (MDC process).
- In Chapter 5 through 10, we switched our attention to completely new areas: advanced technologies that can improve sugarbeet processing and sugar-factory profitability. In Chapter 5, we particularly learn about ion-exchange resin, which is used in most of these technologies used lately in beet-sugar industry.

CHAPTER

ION-EXCHANGE RESIN 5

SECTIONS

- Ion-Exchange Resin Terminology
- Resin Loss and Replacement

ABOUT THE CHAPTER **Chapter 5** is a simplified overview of the properties of the **ion-exchange resin** (simply resin). Knowing the basics about resin is a prerequisite to what you learn in Chapter 6 (Juice-Softening Process) and Chapter 8 (Molasses-Desugaring Process).

In previous chapters, you learned how sugarbeets are processed in a conventional sugar factory to produce quality sugar. Now it is time to learn about technologies that can improve sugarbeet processing and efficiency and profitability of the factory. Most of these technologies use resin. Since the 1980s, some resin-based technologies have become increasingly essential to the sugar industry. Soon, one or more of the techniques, which are based on resin technology, will become a standard part of your facility as well. These techniques include thin-juice softening, juice decolorization, Quentin process, Gryluss process, NRS process, and most important, molasses desugaring by chromatographic (MDC) process (Chapter 8).

Although some resin-based techniques considerably improve the profitability of the facility, some sugar technologists (particularly the older class) hesitate to use them. Unfamiliarity with the basics of the ion-exchange resin and fear of learning are the main reasons for their hesitation. However, learning the basics of resin technology is easy, as you will find out by studying the following few pages.

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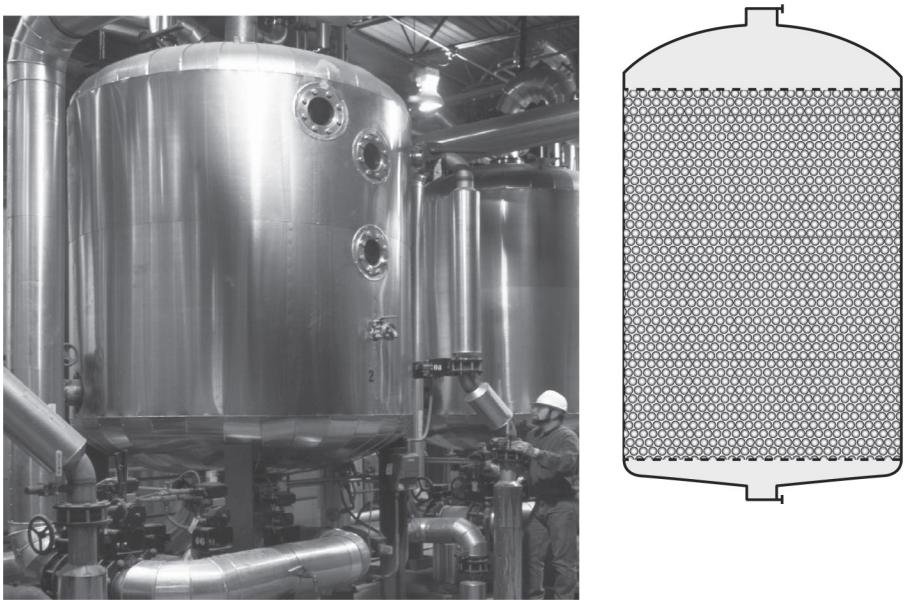


Figure 5.1 A thin-juice softening column (on the left) and resins inside the column (right)

Ion-exchange resin (later resin) is a porous-insoluble polymer in the form of small (1 to 2 mm diameter) particles (**beads** in resin technology). Resin particles are usually yellowish and can operate at high temperatures and different pH ranges. The support structure (**matrix** in resin technology) of resin is like that of sponge, but the holes are too small to be visible to the naked eye. Because of porosity, resin has a very large surface area, so it can selectively exchange ions (cations and anions) in a solution for other ions, hence the name **ion-exchange resin**. The process of exchanging ions for another's by resin (e.g., in juice softening by chromatographic process) and separating components of a compound from each other (e.g., in molasses desugaring by chromatographic (MDC) process) is called **chromatographic process**.

Successful application of resin in the water-softening process has provided the start for its use in other industries. The beet-sugar industry has been using resin for over half a century in different technologies. Juice softening by chromatographic process (discussed in Chapter 6) and molasses desugaring by chromatographic (MDC) process (discussed in Chapter 8) are the most important resin-based technologies used in modern beet factories.

Exchange resins are usually produced by polymerization of **styrene** and a small amount of **Divinylbenzen** (DVB) is added to styrene during the polymerization process. DVB is added for **cross-links** (covalent bonds linking the chains in a polymer) purposes. The DVB content determines many of the chemical and physical properties of the resin, such as the following:

- **Expansion** (the higher the DVB content, the less the resin expands during operation)
- **Moisture content** (the higher the DVB, the greater is the moisture-absorption ability)
- **Capacity** (the higher the DVB content, the higher is the capacity of the resin)
- **Cross-linking** (the higher the DVB content, the stronger is the cross-link)
- **Porosity** (the higher the DVB content, the larger is the pore size)

Ion-exchange resins are divided into two main groups:

- Cation-exchange resins (catex), which remove cations from a solution
- Anion-exchange resins (anex), which remove anions from a solution

Cation resins contain a **functional group** (exchangeable group) that has a negative charge, such as $(SO_3H)^-$ with the ability to remove cations (postitively-charged ions). A functional group is a group of atoms in a molecule that behaves as a unit and is responsible for its reactions with other substances. Examples of functional groups in resins are the sulfonic (SO_3H) and carboxyl (COOH) groups. In the **cation resins**, the **exchangeable ion** of the functional group (e.g., H in SO_3H) can be exchanged for a particular cation present in a solution because of its **affinity** (capability of absorption) for that ion. For example, H^+ can be exchanged for calcium ions (Ca^{2+}) and magnesium ions (Mg^{2+}) in thin juice by passing the juice through a **resin bed** (a small or large column filled with resin). When the juice passes through the resin bed containing the SO_3H group, the following reactions take place:

$$R\text{-}(SO_3H)^- + Ca^{2+} \leftrightarrow R\text{-}SO_3Ca + H^+$$

$$R\text{-}(SO_3H)^- + Mg^{2+} \leftrightarrow R\text{-}SO_3Mg + H^+$$

Here, R indicates the matrix (structure) of the resin; the rest is its functional group. Because of this reaction, hydrogen ions (H^+) from the resin are replaced by the calcium and magnesium in the juice. The treated juice is commonly termed **soft juice** because it no longer has calcium and magnesium. This process has been named **thin-juice softening** (the subject of the next chapter).

Anion resins contain a functional group that has a positive charge with the ability to remove anions (ions with negative charges) from the solution.

The subject gets more interesting when a solution containing cations and anions is passed through a column with a mixture of cation and anion resin, called **mixed-bed resin**. The result is a solution free of cation and anion. You might ask, "Can we treat diffusion juice with cation and anion resin and not use lime during the juice-purification process?" The answer is yes, but the process is much more expensive than the lime method (traditional juice purification). The cation-resin treatment on thin juice (named **thin-juice softening**, discussed in Chapter 6) is provided in some sugar factories for a more complete purification and the production of soft juice.

Cation-exchange resins can be classified based on the behavior of their functional groups:

- Strong-acid-cation resins (SAC resin)
- Weak-acid-cation resins (WAC resin)

Strong-acid-cation resins (SAC resins) are highly ionized in both the acid ($R\text{-}SO_3H$) and salt ($R\text{-}SO_3Na$) form. The functional group of both forms of SAC resins is highly ionized and the exchangeable H^+ and Na^+ are readily available for exchange over the entire pH range. Because of the complete ionization of the functional group of SAC resins, they are called strong resins in the same sense as strong acids (e.g., sulfuric acid) are defined. In the sugar industry, the SAC resin in **Na** form is used in the **juice-softening process** (Ca and Mg removal from the juice) by Gryllus and NRS methods (detailed descriptions are given in Chapter 6).

Weak-acid-cation resins (WAC resins) have a much higher affinity than SAC resins for hydrogen ions (H^+). This characteristic allows for regeneration to the H^+ form with much less acid than is required for SAC resins. In WAC resins, the functional group is a **carboxylic acid** (COOH). Unlike SAC resins, the degree of ionization of WAC resins is strongly affected by the pH of the solution. As a result, resin capacity depends in part on the pH of the solution. In the sugar industry, the WAC resin in H form is used in the juice-softening process by the WAC resin developed by Amalgamated Research Incorporated (see Chapter 6).

ION-EXCHANGE RESIN TERMINOLOGY

Like any other technology, resin technology has its special terms, so some of them will be defined here.

Na^+ form or **H^+ form** are terms often used in ion-exchange technology. For example, when a strong-acid-cation resin with the sulfonic group ($R\text{-}SO_3H$) is placed in a sodium hydroxide solution (NaOH), the resin replaces its cationic part (H^+) for a sodium cation (Na^+) to form $R\text{-}SO_3Na$ (or simply R-Na). When the reaction is complete, the resin is said to be in the sodium form (Na^+ form).

$$R\text{-}H^+ + NaOH \leftrightarrow R\text{-}Na^+ + H_2O$$

Note: Ion-exchange reactions are reversible (the reversible reactions are indicated by a two-way arrow ↔) and stoichiometric. (React based on their equivalent-mass ratio.) In this reaction, the H ions of resin was exchanged with Na ions of the caustic (NaOH) stoichiometrically.

The **selectivity coefficient** (K) is the measurement of a resin's preference for a particular ion. In other words, K determines mathematically the value of a resin's **affinity** (*the larger the K value, the greater is the affinity of the resin for the ion*). The displacement of ions occurs based on the selectivity coefficient. In the preceding example, the selectivity coefficient at equilibrium (at the end of regeneration with NaOH) can be expressed as follows:

$$K_S = \frac{[H^+]_R [Na^+]_S}{[Na^+]_R [H^+]_S} \tag{5.1}$$

In this equation, the R subscript indicates the resin phase, and S indicates the solution phase. The brackets indicate the ion concentration. The concentration of the resin phase is usually expressed in mmol/g, and the concentration of the solution phases is expressed in mmol/ml.

As stated previously, the displacement of ions is based on the resin affinity for particular ions. For example, if resin is in hydrogen form (H^+ form) and is surrounded by a solution containing sodium ions (Na^+ ions), the resin displaces its hydrogen ion for sodium because the resin has greater affinity for the Na^+ than for the H^+. Table 5.1 gives the relative affinity of the cation resin for various ions, compared with the hydrogen ion as standard. (Note: These data are for classical cation resins with high exchange capacity, and the K of other resins might be quite different and lower.)

Exchange capacity of resin (simply capacity of resin) is the ability of resin to remove ions. Resin capacity is usually expressed as gram per liter of resin (g/L). For example, if a resin's capacity is 25 g per liter and it is in Na^+ form, a liter of this resin can displace 25 g cations for sodium. The theoretical column capacity (loading) is defined as the **hardness removal efficiency** (hardness in feed minus hardness in the soft product) multiplied by the theoretical

Table 5.1 Affinity of Cation Resins with Different DVB Content for Various Ions

Cation	*K* 4% DVB	*K* 8% DVB	*K* 10% DVB
H^+	1.0	1.0	1.0
Na^+	1.2	1.6	1.6
K^+	1.7	2.3	2.5
Mg^{2+}	2.2	2.6	2.6
Ca^{2+}	3.1	4.1	4.4
Ba^{2+}	5.7	9.1	9.4

capacity of the resin. The hardness is expressed as CaO (see the Hardness subsection in Section 3 of Chapter 1).

Regeneration of resin is a term used when the resin active part is exhausted, and it must be regenerated (reactivated) from time to time by contact with a regenerant to be converted back to its original activity. When regeneration is complete, the resin gains again its exchange capacity and becomes ready for operation. For example, if the resin was originally in sodium (Na) form and during exchanging (exhausting) step its Na ions were replaced with calcium (Ca) ions from the solution, the resin becomes exhausted with Ca ions and needs regeneration with a regenerant that contains Na ions. In resin work, this action is called **regeneration of resin.**

The life expectancy of resin is defined as that period during which the resin can function effectively. The life expectancy of the resin is affected by its stability against thermal shock, pressure shock, the way it was regenerated, and microorganism growth, as well as treatment of the resin when it is not in use between usages. Microbiological growth is more active if the resin is kept untreated between usages.

Resin fouling is a term used when the resin is fed with feed with a high amount of impurities. Resin capacity decreases when feed contains high-molecular components such as coloring materials.

Resin Leakage (bleed through) refers to partial elimination of separating ions by the resin. To make the ion-exchange process economically feasible, an optimum balance must be reached between the factors affecting the ion-exchange process such as resin-capacity usage and regenerant level usage. This generally means that some ion-leakage is allowed because it is too costly to remove all removable ions.

Resin expansion (swelling) when placed in a solution depends on the percentage of cross-linking (*the greater the number of cross-links, the less the expansion*). Unlike strong-cation resin, weak-cation resin expands significantly at the beginning of the exchanging (exhaustion) step. Resin expands also at the backwashing step. For weak-cation resins, the optimum expansion is 200% of resin volume. (Note: Columns must be designed to allow for expansion.)

Resin particle sizes and their uniformity have considerable effect on separation efficiency. Resins with small particles have a higher exchanging capacity, but the particles must not be smaller than 0.25 mm to avoid high-pressure drop. Usually, a resin size between 0.3 and 0.4 mm is desirable. The **density** of resin particles is about 1.3. The bulk density usually varies between 0.5 and 0.7 g/ml.

NOTE

Resins used in food processing require careful preparation before being used. The resin bed should be flushed with at least three bed volumes (BV) of soft water until the effluent is clear.

RESIN LOSS AND REPLACEMENT

Resins do not stay stable forever, so they have a limited lifetime. Depending on the type of usage, resins are replaced once every several years. Some processes need replacement every year, but others are still in use with no replacement after years. Resin is replaced every 3 to 4 years in thin-juice softening and 6 to 8 years in **molasses desugaring by chromatographic** (MDC) **process**. In addition, the wear-out loss from chemical and mechanical damage ranges from 3 to 15% a year. Operating factors affecting resin lifetime follow:

- **Temperature:** Usually the manufacturer recommends the operating temperature
- **Oxidation:** Some oxidants, such as chlorine and hydrogen peroxide, damage resins
- **Fouling:** Precipitation in resin particles causes fouling, lowering resin life expectancy
- **Osmotic shock:** When resin is exposed to a wide variation in feed concentration or temperatures, it contracts and expands, resulting in cracking and finally breaking the particles

(Note: Resin replacement is expensive, so the system should be operated in a way that minimizes replacement.)

END-OF-THE-CHAPTER NOTE

By studying Chapter 5, you became familiar with the basics of resin technology. This may make you think that resin technology is not as hard as you thought. Now that you have performed the prerequisite study, you are in a position to grasp the subjects in Chapters 6 and 8.

CHAPTER

6

JUICE-SOFTENING PROCESS

SECTIONS

- Soft Juice Produces Soft Molasses
- Juice-Softening by Chromatographic Process
- Juice-Softening Systems
- Juice-Softening by SAC Resin
- Juice-Softening by WAC Resin
- Juice-Softening by Two-Step Process
- Operating Problems

ABOUT THE CHAPTER **Chapter 6** describes different methods of juice-softening processes based on the ion-exchange resin. The chapter covers most of the softening techniques available to the sugar industry, such as the juice softening by SAC (strong-acid-cation) resin and by WAC (weak-acid-cation) resin. Gryllus and NRS juice softening (both use SAC resin) are also covered.

The information given in this and the next chapters helps the reader better understand the molasses desugaring by chromatographic process (**MDC process**), which is detailed in Chapter 8. You cannot learn about resin-based technologies without knowing about the ion-exchange resin. Therefore, before studying this chapter, you should review Chapter 5, which provides a simplified overview of the ion-exchange resin.

The amount of **hardness** (soluble calcium and magnesium salts, also known as **limesalts**) left in the juice after purification has a negative effect on the entire process of sugar production. In addition, the MDC process cannot operate if the molasses fed into the system contains any considerable amount of hardness. Therefore, sugar factories equipped with the MDC process have a thin-juice softening station to remove the final traces of hardness from the juice.

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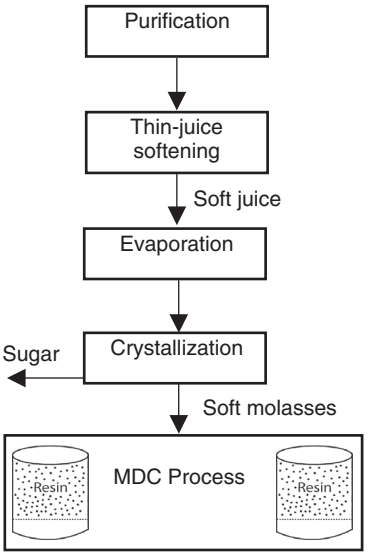


Figure 6.1 Thin-juice softening



Figure 6.2 Thin-juice softening columns

Juice-softening by chromatographic process has been used in the sugar industry since the early 1980s. This process uses resin (ion-exchange resin) to remove the **hardness** (limesalts) left in the **thin juice** after the juice-purification process. **Hardness compounds** (e.g., $CaCO_3$, CaC_2O_4, $MgSO_4$, and $CaSO_4$) are the dissolved salts of calcium (Ca) and magnesium (Mg) that

are not removed during second carbonation. These salts are part of the nonsugars and are not filterable, so they stay with the juice throughout the process. During evaporation, a portion of these salts precipitate in the evaporators (in the form of scale deposits), but the larger portion stays in the juice throughout the process and ends up in molasses.

Thin juice (the product of purification and feed to the evaporation station) always contains a certain amount of hardness. Hardness content of 50 to 200 mg/100 g *DS* (reads milligrams per 100 grams of dry substance) is normal, but it may be more than 600 if the factory processes damaged beets. Hardness is higher also in factories that receive beets from nonirrigated areas or with high alkaline soil. For example, the hardness content is particularly high in countries such as Morocco, Spain, and Iran and in the state of Michigan in the United States. In general, the hardness content in thin juice depends on the following factors:

- Soil in which sugarbeets are grown
- Beet quality and storage condition
- Method of purification operation

Thin juice containing large amounts of hardness, called **hard thin juice**, has some undesirable qualities for sugar processing. When the juice is heated in the heaters and evaporators, the reactions of hardness compounds with other ions become more active, so the positively calcium (Ca) and magnesium (Mg) ions bind with negatively charged ions to form solid compounds. Therefore, the two stations mainly affected by hardness in a negative way in sugarbeet processing are the following:

- **Evaporation:** The solid compounds that have more tendency to precipitate at higher temperatures settle on the tubes causing scale buildup, and consequently reducing heat transfer
- **Crystallization:** The solid compounds cause syrups to become viscous, resulting in low mobility of molecules, slow crystallization, low sugar recovery, and consequently high molasses purity

Following are some major benefits of the juice-softening process:

- Reduces scale formation in evaporators and juice heaters
- Provides a more efficient crystallization and sugar recovery
- Saves on energy and cleaning costs (boilout) of evaporators
- Prepares higher-quality soft thin juice for the rest of the process
- Reduces (or eliminates) the use of antiscalants during evaporation

In addition, cost-effective technologies such as plate evaporators (see Section 13 of Chapter 3) and juice storage (see Section 15 of Chapter 3) can be better justified by the softening process and the elimination of hardness, which otherwise precipitates on evaporator plates or on storage tanks and pumps. In addition, when softening by weak-cation resin is used (more discussion later), the process produces calcium sulfate during regeneration, which can be used as a pulp-press aid in the diffuser, reducing production costs.

Hardness also affects considerably the performance of the factories equipped with the molasses desugaring by chromatographic process as you will find out next.

SOFT JUICE PRODUCES SOFT MOLASSES

Soft juice (refers to soft thin juice), in beet-sugar industry, refers to the juice that can be processed to produce soft molasses. **Soft molasses** refers to molasses with hardness content equal or below 3.0 *mEq*/100 *DS* (reads milliequivalent per 100 g of dry substance). This equates to 85 mg/100 g *DS* or 850 ppm/*DS*, measured as CaO. (The term **measured as CaO** is explained in the Notes on page 494.)

To convert hardness content expressed in mg to *mEq*, and vice versa, you can use equivalent mass formula (Eq. 12.5 given in Chapter 12). The equivalent mass (*Eq*) is based on the molecular mass (*MM*) of the substance and its number of charges (N_C):

$$Eq_{CaO} = \frac{MM}{N_C} = \frac{56}{2} = 28 \qquad \frac{85\,mg}{28} = 3.0\,mEq$$

Molasses containing more hardness than 3.0 *mEq*/100 *DS* is known as **hard molasses** (standard molasses). If hard molasses is fed to the **separator** (separation column filled with resin) of the MDC process, the resin becomes fouled with calcium and magnesium salts and will soon lose its ability to separate sucrose efficiently. To prevent resin fouling, the separator should be fed with soft molasses. Soft molasses can be produced by two methods:

- Thin-juice softening process
- Molasses-softening process

In the first method, the thin juice is decalcified by resin before the evaporation process. In the second method (the subject of the next chapter), molasses is chemically decalcified. The first method provides the advantage of eliminating scaling in the evaporators and improving sugar recovery, while also providing soft molasses for using in the MDC process. Because the operating costs of both methods are almost the same, the more benefits gained from the first method make it more attractive than the second method (see the following Note).

NOTES

- **Factories equipped with juice softening and molasses desugaring by chromatographic process (MDC process) need both softening systems if they obtain part of their molasses from sources that produce hard molasses.**
- **Sugar scientists believe that thin-juice softening is a positive process in conventional sugar processing, even when the factory is not equipped with the MDC process.**

The **hardness** (limesalts) content in thin juice and molasses is usually expressed as mg in 100 g of dry substance or mg in 100 g of juice (on juice). For example, thin juice with 150 mg/100 *DS* contains 150 mg of hardness in 100 g of its *DS*. Suppose that the *DS* content of the juice is 15%. Then, this would equate to 22.5 mg (150 × 0.15 = 22.5) on juice.

The hardness content of the juice determines to what level it has to be softened to produce soft molasses. An equivalent relationship between the amount of hardness in the thin juice (HS_J) and in molasses (HS_M) must be established. This can be achieved from the ratio of nonsugar (nonsucrose) content of the juice (NS_J) and molasses (NS_M):

$$HS_M = HS_J \times \frac{NS_M}{NS_J} \tag{6.1}$$

$$HS_J = HS_M \times \frac{NS_J}{NS_M} \tag{6.2}$$

Some sugar technologists do not express hardness content in the thin juice and in molasses in *identical units*. In this case, you can use the following equations for an equivalent relationship between the two units:

$$HS(mEq) = \frac{HS(mg)}{28} \tag{6.3}$$

$$HS(mg) = HS(mEq) \times 28 \tag{6.4}$$

(Note: The equivalent mass of CaO is 28, as was calculated earlier.) Study the following example:

EXAMPLE 1

Given:

Thin-juice hardness after softening	25 mg/100 *DS*
Thin-juice purity	90.0%
Thin-juice *NS*	100–90.0 = 10.0%
Molasses purity	60.0%
Molasses *NS*	100–60.0 = 40.0%

Calculate:

Hardness in molasses produced from this juice

Whether this juice is soft enough to produce soft molasses

Equation (6.1) calculates the hardness content of molasses:

$$HS_M = HS_J \times \frac{NS_M}{NS_J} = 25 \times \frac{40.0}{10.0} = 100\,\text{mg}$$

or

$$HS(mEq) = \frac{HS(mg)}{28} = \frac{100}{28} = 3.6\,mEq$$

In this case, molasses is not soft enough (it has more than 3 mEq hardness) to be used in the MDC process. Equation (6.2) calculates the extent to which the juice must be softened to produce soft molasses:

$$HS_J = HS_M \times \frac{NS_J}{NS_M} = 3.0 \times 28 \times \frac{10.0}{40.0} = 21\,\text{mg}/100\ DS$$

This means that the juice has to be softened until its hardness content equals 21 mg to produce a molasses with 3 *mEq* hardness.

NOTES

- **Based on the preceding calculations, one can state that thin juice (with about 15% *DS*) containing less than about 20 mg/100 *DS* hardness can be considered soft juice. Soft juice is further processed to produce soft molasses, which is required when a factory is equipped with the MDC process.**
- **It is not economical for the sugar factory to soften thin juice with a hardness less than 17 mg/100 *DS* (or 17 × 15/100 = 2.5 mg/100 ml juice or 25 ppm). Therefore, in the juice-softening operation, operators try to maintain a juice hardness between 13 and 20 mg/100 *DS* to achieve a juice with an average hardness of about 17.**
- **Terms such as measured as CaO (or as CaO) or sometimes "measured as $CaCO_3$" are used in the softening process. As previously stated, hardness consists of a number of calcium and magnesium salts in the sample. If each salt were reported individually (e.g., $CaSO_4$ as $CaSO_4$, MgO as MgO), it would be difficult to add all the results together. To simplify calculations and laboratory analyses, the total number of all hardness salts is reported in terms of "as CaO." This establishes an equal denominator for all components, resulting in a direct relationship among the amounts of different components present in the sample and expressing the total amount as one component. Further information on the calculation is at the end of this chapter in the Calculation of Soda Ash Dosage section.**
- **In the laboratory, chemists prepare a standard acid solution (an acid solution with an exact concentration) based on the equivalent ratio of acid to CaO. Then, the hardness is determined from a titration of a measured volume of a juice sample against the standard acid. The result indicates the total hardness of the sample as CaO.**

JUICE-SOFTENING BY CHROMATOGRAPHIC PROCESS

Juice-softening by chromatographic process is a separation technique based on the exchange of ions by ion-exchange resin (simply resin). In this process, the resin in a column selectively attracts some ions and releases other ions, so the process is an exchange operation, hence the name **ion-exchange chromatography**. The process is similar to water softening in households, which involves the exchange of hard-water cations of Ca^{2+} and Mg^{2+} for sodium cations (Na^{+}) from resin to soften water. When resin becomes nearly exhausted with Ca and Mg ions, it should be regenerated with a solution containing sodium, such as salt solution (NaCl).

In a typical thin-juice softening by chromatographic process (see Figure 6.3), thin juice is fed to large softening columns that are filled with resin. The softening column is 3 to 5 m high and 2 to 3 m in diameter (see Figure 6.2). As the juice passes over the resin, calcium (Ca^{2+}) and magnesium (Mg^{2+}) ions displace the ions from the resin (usually sodium or hydrogen ions in thin-juice softening). For example, when resin is in Na form, Ca and Mg ions in the juice are displaced by Na ions. The Ca and Mg ions can be displaced because their positive charges (2+) are greater than that of the Na ions (1+). In other words, Ca and Mg have greater affinity (attraction ability) for the negative sites on the resin (the matrix of resin with negative charge) than sodium.

Based on the chemistry of the juice-softening process, for every ion that binds with the resin, two Na ions from the resin are released to the juice. Therefore, the juice that exits the softener (few columns filled with resin and accessories) is almost free of Ca ions but contains Na ions in their place. The liquid leaving the softener is called the **soft juice** because Na ions are much less **scale-forming** than calcium. Eventually, the resin is filled with calcium cations. At this time, the resin needs to be **regenerated** (reactivated). The flow of the feed to the column stops and the resin regeneration cycle starts. The flow is switched to the next column (called standby column), which is already regenerated.

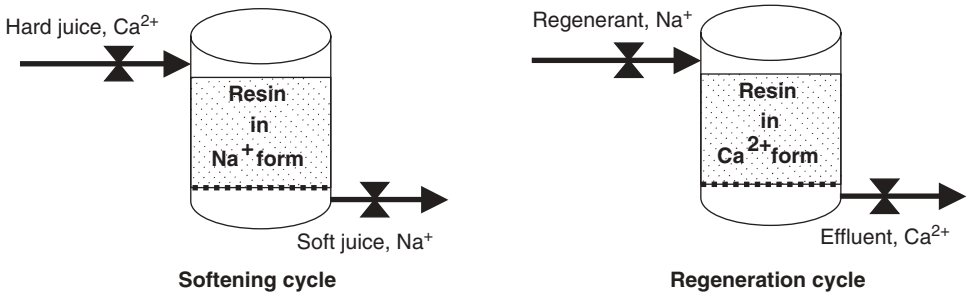


Figure 6.3 A typical thin-juice softening process

Resin is regenerated by pumping a **regenerant**, which is a solution containing Na ions (usually diluted NaCl or NaOH), to the softener column. During regeneration, calcium ions are removed from the resin and sodium ions occupy their place in the resin. After regeneration, the resin is ready again for the next softening cycle.

JUICE-SOFTENING SYSTEMS

Several juice-softening systems have been installed in beet-sugar factories to supply softer juice for processing. The process of juice purification followed by juice softening can eliminate more than one-third of the nonsucrose content present in the juice. Therefore, juice softening can be considered part of the purification station and its efficiency enhancer.

Few methods are available for thin-juice softening. Some methods, such as the **Gryllus process**, partially soften the juice, which is good enough for only conventional factories mainly because they cannot produce molasses of sufficiently low hardness for direct feed to the MDC process. Some systems, such as the **NRS** (new regeneration system), provide both the advantage of juice softening and the production of soft molasses that can be used in the MDC process. Therefore, the selection of the softening method is important, particularly when the factory is equipped with the MDC process.

In the juice-softening process, a cation resin (either strong or weak) is used to selectively exchange some cations of the juice. An understanding of the differences in properties between **strong-acid-cation** (SAC) **resin** and **weak-acid-cation** (WAC) **resin** is important because both are used in thin-juice softening. The major differences follow:

- SAC resins have a lower exchange capacity than WAC resins.
- SAC resins (in Na form) provide no risk of sucrose inversion, while WAC resins (in H+ form) may cause some sucrose inversion.
- SAC resins operate at a lower flow rate (because of a lower exchange capacity) than WAC resins, which operate at 50 to 100 **bed volumes** (BV) per hour.
- SAC resins remove all cations, whereas WAC resins remove only cations equivalent to bicarbonate alkalinity (natural alkalinity).
- SAC resins create some dilution during operation, whereas WAC resins create more dilution.
- SAC resins expand less during regeneration than WAC resins, which expand almost twice when they are exhausted.

- SAC resins need a more excessive regenerant over the **stoichiometric quantity** (SQ) for regeneration (50 to 150% over SQ) than WAC resins (about 10% over SQ).

Although the advantages are in favor of SAC resins, a softening system that uses WAC resin is also used successfully in some U.S. beet factories, as explained later. Operation of the softening process generally consists of the following cycles:

1. **Service:** Heated juice (80 to 90°C) is passed continuously through the resin bed until the resin is nearly exhausted (saturated with the hardness). (The higher amount of hardness in the treated juice signifies that the resin is exhausted.)
2. **Backwashing:** The resin is backwashed with soft water (factory condensate) to reclassify the resin particles and to remove particulates. The backwashing cycle brings the resin to its original order because, during the service cycle, resin particles become classified (the largest particles remain at the bottom, while the smaller ones are distributed on a higher level). A backwash is provided by applying a uniform flow of water from the bottom of the bed to fluidize the resin. Typically, the volume of water required is about 2 BV. To backwash the resin correctly, the resin level should be expanded to within 0.3 m (2 ft) of the distributor at the top of the column.
3. **Regeneration:** The resin is regenerated with a regenerant to return the resin to its original capacity.
4. **Rinse:** The resin is rinsed with soft water.
5. **Service:** The regenerated column is again fed with juice.

Typical position of in and out of the column during steps follows:

Steps	Flow In	Flow Out
Sweet-on	Hard juice	Soft juice
Sweeten-off	Condensate	Soft juice
Backwash 1	Condensate	Condensate
Regeneration	Regenerant	Regeneration effluent
Rinse	Condensate	Regeneration effluent
Backwash 2	Condensate	Condensate
Stand-by		

Note: There is no sweeten-off or sweeten-on step in the NRS system because only soft-thin juice is used in the backwashing, regeneration, and rinse steps. During regeneration, caustic is added to the soft-thin juice to perform the regeneration.

The size of the columns depends on the amount of feed to be processed and its hardness content. Because the regeneration of the resin beds requires twice as much time, you need double the number of columns. During operation, usually half of the columns are in exchange position, and the other half undergo regeneration, or stay on standby for a new use. Typical positions of the columns, based on the design, are as follows:

Three-column design	Two in exhausting and one in regenerating or on standby
Four-column design	Two or three in exhausting and one or two in regenerating or on standby
Five-column design	Three in exhausting and two in regenerating or on standby

Nowadays, softening systems are fully automatic and computer controlled. A flowmeter and flow-control valve on each column regulates the flow through each step of the operation.

The two different softening methods often used in sugar factories are

- Juice-softening by strong-acid-cation (SAC) resin
- Juice-softening by weak-acid-cation (WAC) resin

Both methods are described in the following section.

JUICE-SOFTENING BY SAC RESIN

In the juice-softening by **SAC** (strong-acid-cation) resin, the softener uses resin mainly in sodium form (Na^+) to soften the juice. When the juice containing divalent cations (Ca^{2+}, Mg^+) passes through the resin, the following reactions take place:

$$2R - Na^+ + Ca^{2+} \leftrightarrow R_2 - Ca + 2Na^+$$

$$2R - Na^+ + Mg^{2+} \leftrightarrow R_2 - Mg + 2Na^+$$

Scale-forming cations of the juice (Ca and Mg) are replaced by non-scale-forming sodium ions from the resin. The efficiency of replacement determines whether the juice is soft enough to produce soft molasses. Besides type of resin, the efficiency depends on the type of resin regeneration. Based on the type of regeneration, thin-juice softening by strong resin can be divided into three groups:

- **Conventional method:** Uses Na^+ ions in NaCl solution for regeneration
- **Gryllus method:** Uses Na^+ and K^+ ions in low-green syrup for regeneration
- **NRS method:** Uses Na^+ ions in NaOH solution and soft juice for regeneration

Conventional Juice Softening

In **conventional juice softening**, the softener uses common salt (NaCl) in a 10% solution to regenerate the resin. Salt is used mainly because it is inexpensive and plentiful. The process is similar to household water softening, which uses resin to remove the hardness from the water.

About 200 g of salt per liter of resin is used for regeneration. For an average hardness content of 150 mg/100 *DS*, the capacity of the resin is about 20 g CaO per liter of resin. Therefore, salt usage is 200/20 or 10 g per g of removed CaO (Lancrenon 1984).

Following are disadvantages of the conventional method:

- High water use
- High wastewater
- High sugar loss (Na is more melassigenic than Ca)
- High juice dilution (because after sweeten-off and regeneration, the resin is rinsed with water)

Gryllus Juice Softening

In **Gryllus juice softening** (named after the Hungarian inventor, Eva Gryllus), ions of Na and K in the **low-green** (C) **syrup** are used to regenerate the resin (see Figure 6.4). High sodium and potassium in the syrup make it very efficient to regenerate the resin after exhaustion with calcium (Gryllus et al. 1998).

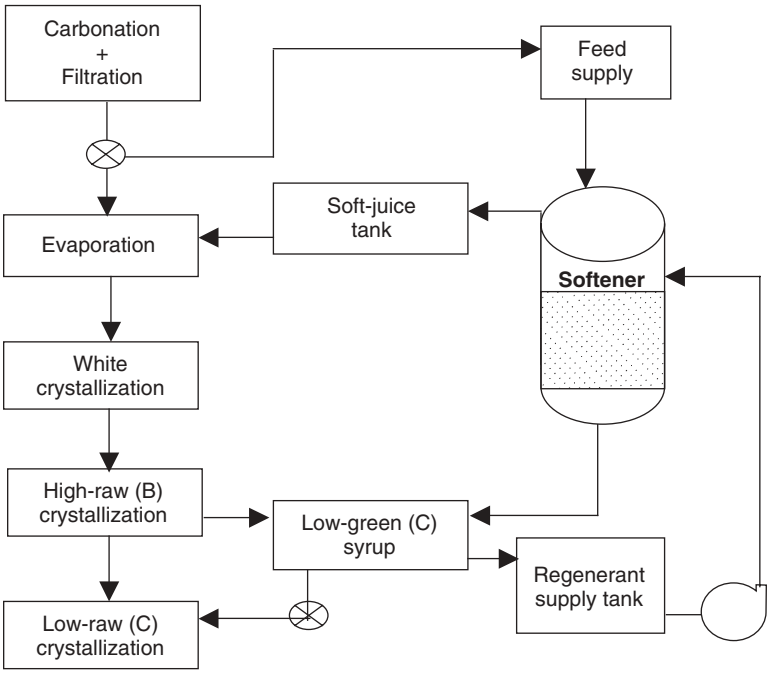


Figure 6.4 Flow diagram of Gryllus juice softening

The Gryllus method eliminates the main disadvantage of the conventional method by not producing wastewater. During regeneration, effluent is sent to **low-raw** (C) **crystallization** (the third stage of crystallization). Therefore, the hardness substances bypass part of the processing steps (evaporators, white crystallization, and high-raw crystallization) and end up in molasses. The practice of bypassing the hardness is called the **Gryllus effect**.

Compared with the conventional process, the Gryllus method has the following advantages:

- No wastewater
- No juice dilution
- Clean evaporators
- Better fuel economy
- No change in the melassigenic properties of the nonsucroses

The disadvantages of the Gryllus method are as follows:

- The juice is not soft enough to produce soft molasses; therefore, the process *is not suitable for factories that have (or plan to have) the molasses-desugaring process.* (Note: Molasses leaves at the same hardness as the no-softening process because the hardness bypasses only the evaporators, first- and second-stage of crystallizations, and ends up in molasses.)
- It works well only where limesalts are relatively low (below 200 mg/100 *DS*).
- It depends on the amount of low-green syrup because the alkali ions in the juice should be enough to regenerate the resin.

- It depends on the Na concentration in the beets being processed and the use of soda ash in the process.

NRS Juice Softening

In **NRS** (new regeneration system) juice softening, Na^+ cations in caustic soda (NaOH) solution and in the soft juice are used to regenerate the resin. This process does not have the disadvantages associated with the conventional process (NRS does not produce *waste effluent*) and the Gryllus process (NRS can soften juice to the extent that *soft molasses can be produced*.)

In NRS softening (see Figure 6.5), soft-thin juice is cooled to 40°C and mixed with 50% caustic. The mixture is then used to regenerate the resin. The regeneration is operated at a low temperature to produce **soluble calcium saccharate** (mono- and di-calcium saccharates are soluble in water). During regeneration, the sodium from the caustic is exchanged for the calcium ion in the resin. At the same time, sucrose reacts with released calcium ions to form soluble calcium saccharate. Regeneration effluent is pumped to the first-carbonation tank, where CO_2 decomposes the saccharate into sucrose and calcium carbonate ($CaCO_3$). The second-carbonation juice is filtered and sent to the softener to produce soft juice. A small portion of soft juice is sent back to the softening station to regenerate the resin, while the main part enters the evaporators. NRS softening does not need a sweeten-off or sweeten-on step.

In NRS softening, the process is completed in two steps. The first step takes place in first carbonation, where Na ions displace Ca ions. The source of sodium for this displacement is from the caustic (used at above the stoichiometric amount) in the regeneration stage, which subsequently returns to the carbonation. The second step takes place in the softener containing SAC. Approximately 60% of the softening occurs in the softener and the remaining softening occurs in the carbonation. The overall efficiency of NRS softening is about 96% of the total hardness present in the juice (Rhoten 1995).

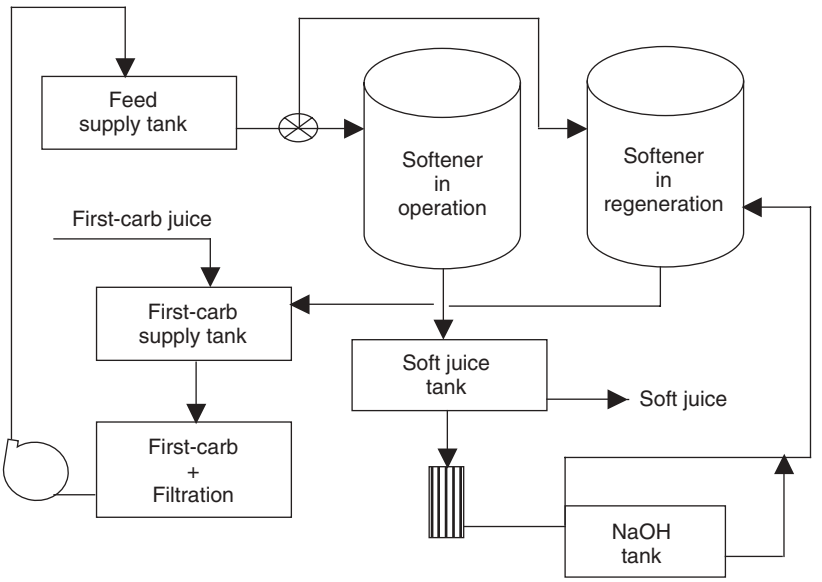


Figure 6.5 Flow diagram of NRS juice softening

The amount of caustic soda used is equal to 40 kg per cubic meter of resin. The required amount of regenerant is about 100% on bed volume (resin volume). The advantages of NRS softening are as follows:

- Produces soft molasses
- No sucrose inversion
- No juice dilution
- No wastewater

The disadvantage of this process is that additional sugar loss occurs because of an increase in molasses production.

JUICE-SOFTENING BY WAC RESIN

The juice-softening by WAC (weak-acid-cation) resin used in the sugar industry was first patented by Amalgamated Sugar (Schoenrock et al. 1975). This technology uses WAC resin, such as Dowex MAC 3, in the hydrogen (H^+) form. Softening occurs according to the following equation:

$$2R - H^+ + Ca^{2+} \leftrightarrow R_2 - Ca + 2H^+$$

When the resin becomes exhausted with calcium, it is regenerated with a low-concentration acid solution. Regenerating the resin with a sulfuric acid solution (H_2SO_4), instead of hydrochloric acid (HCl), has two advantages:

- H_2SO_4 does not corrode stainless steel, while HCl adds chlorides to the system, which corrodes stainless steel and is more melassigenic than sulfate
- H_2SO_4 produces calcium sulfate, which can be used in the diffuser as a **pressing aid** for improving pulp pressability (see Sections 7 and 8 in Chapter 3):

$$R_2 - Ca + H_2SO_4 \leftrightarrow 2R - H^+ + CaSO_4$$

Because calcium sulfate is insoluble at higher concentration, there is a risk of calcium sulfate precipitation inside the resin bed during regeneration. This will result in deposits of calcium sulfate within the resin structure, which fouls the resin. Therefore, to avoid precipitate formation and resin fouling, keep the strength of sulfuric acid low. It is recommended to use H_2SO_4 concentration in the range of 0.07 *N* (about 0.2% acid–see Example 29 of Chapter 12) at 110% on resin volume (Henscheid et al. 1990).

The hot juice should be cooled before it is fed into the column to avoid damage to the resin and to minimize any sucrose inversion. During the first few minutes of the softening cycle in which the juice passes through the resin bed, hydrogen ions are displaced by Na and K. Therefore, no additional Na or K would be added to the juice. During this period, the low pH value of about 3 is present because Ca and Mg are not displaced for H ions. Then, the displacement of Ca and Mg starts, which increases the pH to about neutrality. Approximately 80% of the total resin capacity is taken by the divalent ions, and the remaining 20% is taken by the Na, K, and H ions (Henscheid et al. 1990).

The low pH juice must be adjusted as soon as it comes out of the column to minimize sucrose inversion. **Caustic soda** (NaOH) is usually used to increase the juice pH to 9. The amount of caustic needed to adjust the juice pH can be calculated according to the following example:

EXAMPLE 2

Given:

Juice pH leaving the softener	3
Juice pH adjustment required	9
Juice dry substance (*DS*)	15%
Juice density	1.06 kg/L
Diffusion draft	110%
Caustic soda (NaOH) concentration	50%
Density of 50% NaOH	1.5 kg/L (12.7 lb/ga)

Calculate:

Required amount of 50% caustic soda (NaOH) per t beets processed

The amount of caustic needed to adjust the juice pH from 3 to 9 can be calculated from the initial and final concentration of H^+ in the juice:

$$[H^+]_{Initial} = 10^{-3} = 1 \times 10^{-3} \text{ mole}$$

$$[H^+]_{Final} = 10^{-9} = 1 \times 10^{-9} \text{ mole}$$

At a **draft** (the mass of diffusion juice drawn from the diffuser per 100 kg of beets) equal to 110%, the factory produces 1.1 kg (or tons) of juice per each kg (or ton) of processed beets. The amount of juice (in liters) produced from each ton of beets is

$$1.1t \times 1000 \frac{\text{kg}}{t} \times \frac{1}{1.06} \frac{L}{\text{kg}} = 1038 \text{ liters}$$

Using the volume of the juice, we can calculate the number of moles (M) of H^+ at both pH values:

$$\left(1 \times 10^{-3} \frac{\text{mole}}{\text{L}}\right) \times 1038\text{L} = 1.038 \text{ mole}$$

$$\left(1 \times 10^{-9} \frac{\text{mole}}{\text{L}}\right) \times 1038\text{L} = 1.038 \times 10^{-6} \text{ mole}$$

Therefore, the change in H^+ is

$$1.038 - 1.038 \times 10^{-6} = 1.038 \times 10^{-5}$$

NaOH is very basic. In the neutralization reaction, one mole of OH reacts with one mole of H to form H_2O. Therefore, 1.038×10^{-5} mole of H^+ requires the following number of grams of NaOH:

$$(1.038 \times 10^{-5} \text{ mole } H^+)\left(\frac{1 \text{ mole NaOH}}{1 \text{ mole } H^+}\right)\left(\frac{40.0 \text{ g NaOH}}{1 \text{ mole NaOH}}\right) = 4.15 \times 10^{-4} \text{ g}$$

The amount of 50% NaOH is

$$4.15 \times 10^{-4} \times 1000 \times \frac{100}{50} = 0.8 \text{ kg/t beet}$$

$$\frac{0.8}{1.5} = 0.5\text{L/t beet (or 0.14 ga/t beet)}$$

NOTES

- **The WAC resin expands (swells) considerably (about double) during the first period of sweetening-off because the hydrogen form changes to the monovalent form. Resin expands also during the regeneration stage. Softener columns must be designed to allow for this expansion.**
- **To avoid corrosion, stainless steel (or a suitable alternative) is used in the construction of all areas exposed to acid. Increased corrosion to pipes and evaporators can occur due to the production of soft juice. As a precaution, the evaporators can be coated up to the liquid surfaces.**

JUICE-SOFTENING BY TWO-STEP PROCESS

The **juice-softening by two-step process** was recommended by Amalgamated Research (the supplier of juice softening by WAC resin). It is used mainly when the juice contains high hardness. The process consists of the following two steps:

- **Step 1:** Pretreatment with soda ash to reduce the amount of hardness to the 100 to 150 mg level
- **Step 2:** Treatment with the WAC resin to reduce the hardness to the desired level of about 17 mg (this step is the same as described under the softening by WAC resin section).

The pretreatment relieves the softener of extra load and allows the softener to act as a finishing process. In the pretreatment step, soda ash (Na_2CO_3) is mixed with the *second-carb juice* in such an amount that brings the hardness to the 100 to 150 level. As a result, the Na in the soda ash precipitates (↓) the Ca and Mg in the juice to produce calcium carbonate, which is then separated by filtration before the juice goes to the second step of operation:

$$Na_2CO_3 + CaSO_4 \rightarrow CaCO_3\downarrow + Na_2SO_4$$

$$Na_2CO_3 + MgSO_4 + Ca(OH)_2 \rightarrow CaCO_3\downarrow + Mg(OH)_2\downarrow + Na_2SO_4$$

$$Na_2CO_3 + Ca(CH_3COO)_2 \rightarrow CaCO_3\downarrow + Na_2(CH_3COO)_2$$

Generally, adding soda ash to the juice has the following advantages and disadvantages:

- Decreases the hardness (advantage)
- Increases juice alkalinity (advantage)
- Completes the lime reaction (advantage)
- Increases Na in the juice, which is partly removed by the resin (disadvantage)
- Soda ash is highly melassigenic; Na is more melassigenic than all cations of the juice except potassium; the melassigenic factor of soda ash is 0.42, which is high (disadvantage)

NOTES

- **The chemistry of softening by soda ash is complicated because of the reaction of Na with different nonsugars. For example, unlike lime that reacts with the bicarbonate hardness of the juice, the soda ash mostly reacts with other salts such as the sulfate hardness.**
- **Molasses production and molasses purity are affected not only by nonsugars present in the beet juice but also by the nonsugars added during the process (e.g., in juice purification and softening processes). Soda ash is a strong melassigenic substance (molasses-maker substance). Therefore, when adding soda ash, care should be taken to not add more than required. At high dosage, sodium**

cations (Na^+) become by far the major (in terms of quantity) and the most melassigenic (in terms of quality) nonsugar in the juice. This affects the equilibrium of the nonsugars and its composition per melassigenic value, pH, and alkalinity. Most important, it increases molasses production and molasses purity, which are the most damaging factors affecting the profitability of any beet-sugar factory. (Remember: *Soda ash treatment at the* right dosage *is a benefit to the process, but its* high dosage *damages the process considerably.*)

- **At second-carbonation pH (9.2 at 20°C), most of the alkalinity is in the bicarbonate form, which contributes to the softening process a little. Therefore, adding too much soda ash to the juice is inefficient. In other words, at high dosage, the hardness reduction becomes almost independent of the rate of soda ash addition, and ion-exchange softening must be employed to decrease the hardness to the desired level. This is the reason that two-step softening is used when hardness content is too high.**
- **Amalgamated Research Inc. offers fractal technology to the sugar industry for distribution feed to the softener. The supplier represents economic data for fractal-weak-cation-resin softener, which decreases the payback to two to three years (Kochergin and Tzschatzsch 2005).**

Calculation of Soda Ash Dosage

Because one part of soda ash reacts with one part of calcium and magnesium salts, the required dose of soda ash is equal to the desired reduction of the hardness. To remove hardness in each kg (or ton) of juice, the required dose of soda ash is based on the relative **equivalent masse**s (*Eq*)of soda ash and the total masses of $CaSO_4$, $MgSO_4$, and $Ca(CH_3COO)_2$ (measured as CaO). The following example clarifies the subject:

EXAMPLE 3

Given:

Juice hardness before soda ash addition	200 mg/100 *DS* as CaO
Desired hardness after soda ash addition	100 mg/100 *DS* as CaO
Juice dry substance (*DS*)	15%
Draft	115%

Calculate:

Soda ash dose required (% on juice and on beet)

To calculate hardness on solid bases, the hardness (as CaO) in the juice should be calculated:

$$100 \times 15\% = 15\,\text{mg CaO}/100\,\text{g juice} \quad \text{or} \quad 0.15\,\text{g/kg}$$

To convert the amount of a substance to its equivalent "as CaO," the amount of substance (in this case, 0.15 g) must be multiplied by the ratio of the equivalent mass (*Eq*) of CaO to the equivalent mass of the substance:

$$0.15\,\text{g (as CaSO}_4\text{)} = 0.15 \times \frac{Eq_{\text{CaO}}}{Eq_{\text{CaSO}_4}} = 0.15 \times \frac{28}{136/2} = 0.06\,\text{g (as CaO)}$$

$$0.15\,\text{g (as MgSO}_4\text{)} = 0.15 \times \frac{28}{Eq_{\text{MgSO}_4}} = 0.15 \times \frac{28}{104/2} = 0.08\,\text{g (as CaO)}$$

$$0.15\,\text{g [as Ca(CH}_3\text{COO)}_2\text{]} = 0.15 \times \frac{28}{Eq_{\text{Ca(CH}_3\text{COO)}_2}} = 0.15 \times \frac{28}{158/2} = 0.05\,\text{g (as CaO)}$$

$$0.15\,\text{g (as Na}_2\text{CO}_3\text{)} = 0.15 \times \frac{28}{Eq_{\text{Na}_2\text{CO}_3}} = 0.15 \times \frac{28}{106/2} = 0.08\,\text{g (as CaO)}$$

$$\text{Na}_2\text{CO}_3 \text{ needed} = \frac{0.08}{0.06 + 0.08 + 0.05} = 0.42\,\text{g/kg of juice}$$

At draft equal to 115%, a factory produces 1.15 kg (or tons) of juice per each kg (or ton) of beets:

$$1.15 \times 0.42 = 0.48\,\text{g/kg of beet or kg/t of beet}$$

Because reactions do not go to completion, soda ash in excess of the stoichiometric amount is needed. Therefore, the amount of soda ash can be increased by 10%:

$$0.48 \times 1.1 = 0.5\,\text{kg/ton beet}$$

This means that the amount of required soda ash to bring the limesalt content down by 100 mg, is equal to 0.5 kg (or 1.1 lb) per ton of beets processed.

NOTES

- **The explanations in this chapter covered the hardness removal (removing Ca and Mg hardness) but not total demineralization (removing Ca, Mg, Na, and K hardness). In the total demineralization process, juice is treated first with the cation-exchange resin in hydrogen form (H^+) or sodium form (Na^+) and then with the anion-exchange resin in hydroxide form (OH^-) or is treated in one stage in a mixed-bed resin. The total demineralization process is not very popular in the beet-sugar industry because of high operation costs, risk of sucrose inversion, and operational complexity.**
- **If the factory has a softening station, the sulfitation (the process of adding sulfur dioxide to the juice) should be performed after the softening process. This is because the sulfur dioxide (SO_2) reacts with cations of the juice to form the sulfates, which give the softener extra work (to remove the sulfates).**

OPERATING PROBLEMS

This section describes solutions to some chemical and operational **problems** that can occur with resins or during operations.

Low Resin Capacity

The usual causes of low resin capacity follow:

- **Improper regeneration:** The concentration of the regenerant might be wrong.
- **Insufficient backwashing:** An insufficient backwashing causes **solution channeling** through the resin bed, lowering the resin capacity. Insufficient backwashing can also cause the incomplete removal of fine particles from the resin, also lowering the resin capacity.

- **Contaminated resin:** Microbial growth in resin, caused by trace amounts of sugar or other organic substances remaining in the resin matrix, can reduce the resin capacity. Microbes are more active if the resin is kept untreated between usages (e.g., between two campaigns). After each campaign, resin should be rinsed thoroughly (2 to 3 BV), double regenerated, rinsed again, and kept in a 10% common salt solution until the start of the next campaign. This type of treatment protects the resin against microbial growth and frost. If contamination occurs, use sodium hypochlorite or hydrogen peroxide to clean the resin. For sodium hypochlorite (KClO), use a concentration of 0.1 to 0.2% (pH 8.8) at about 0.5 bed volumes (**BV**) with a contact time of about 20 minutes, followed by a 2 BV rinse and double regeneration to convert the resin into the appropriate form. For hydrogen peroxide (H_2O_2), use a 0.1% solution at 1 BV with a contact time of about 30 minutes, followed by a 2 BV rinse and double regeneration to convert the resin into the appropriate form.
- **Fouled resin:** Under normal service conditions, low resin capacity may be associated with a resin matrix fouled with a precipitation of calcium sulfate. This problem can be avoided by using the proper sulfuric acid regeneration procedure. An increase in colorants (coloring materials) in the juice can also foul the resin because of the colloidal nature of the colorants.
- **Dry resin:** If the resin gets dry, it can be hydrated with a saturated sodium chloride. Generally, ion-exchange resins should be stored tightly in plastic bags to retain moisture.

High-Pressure Drop across the Column

Resin bed contains numerous but relatively small passages for the feed to travel through the bed. Therfore, the pressure drop throught the bed is critical. A sudden pressure drop across the column (differential pressure) is an indication of a problem. The problems may be easy to solve or may ultimately shutdown the system for corrections. The following are causes of an unusual pressure-drop fluctuation:

- **High viscosity feed:** A viscous juice creates a high-pressure drop.
- **Wrong feed rate:** The flow rate should be based on the concentration, purity, and hardness content of the feed. These factors should be monitored to maintain optimum pressure drop.
- **Insufficient backwashing:** Insufficient backwashing prevents reclassification of resin particles and this creates a high-pressure drop across the resin bed. Backwashing must be performed properly after each cycle.
- **High dry substance feed:** A juice with high dry substance (*DS*) creates a high-pressure drop.
- **High gas content:** A juice with high gas content and bubbles creates a high-pressure drop. Such a feed must be degassed to remove air bubbles to prevent flow channeling.

Complete Loss of Resin Capacity

When a resin completely loses its capacity, it cannot be renewed.

END-OF-THE-CHAPTER NOTES

- **The two main processes for softening thin juice in the beet-sugar industry are the NRS (new regeneration system) and WAC (weak-acid-cation) process. Applexion markets the NRS system and Amalgamated Research Incorporated (ARi) markets the WAC process. Both systems are used throughout Europe and the United States. Beet molasses produced using these processes can be desugared in the MDC process with satisfactory results.**
- **For more information about how to compare different softening systems offered by different suppliers, refer to the Economy of the MDC Process section of Chapter 8.**
- **Sugar technologists, whose factories have the MDC process and purchase hard molasses, should pay particular attention to the next chapter (Chapter 7) because hard molasses has to be softened before it is fed to the MDC process (Chapter 8)**

CHAPTER 7

MOLASSES-SOFTENING PROCESS

Written by C. D. Rhoten

Adapted by M. Asadi

SECTIONS

- Measurement of Hardness in Molasses
- Assessment of Parameters of Molasses Softening
- Continuous Molasses Softening
- Calculation of Chemical Requirements
- Process Control
- Operating Problems

ABOUT THE CHAPTER **Chapter 7** describes the details of the molasses-softening process, which is a new technology in the beet-sugar industry. The information provided here is based on the experiments gained over the last few years during molasses-softening operation in a U.S. beet-sugar factory.

Molasses desugaring by chromatographic process does not operate well if the hardness (limesalts) content of the molasses to be fed to the system exceeds the limit (3.0 *mEq* CaO/100 *DS*), typically referred to as **hard molasses**. If hard molasses is fed to the chromatographic column (**separator**), the resin in the system soon loses its ability to separate sugar efficiently, because of the fouling of the resin with calcium and magnesium cations (hardness). To prevent such fouling, the separator should be fed with **soft molasses** (molasses with a hardness content of 3.0 *mEq*/100 *DS* or less). Soft molasses can be produced by two methods: thin-juice softening (explained in the previous chapter) and molasses softening, discussed here.

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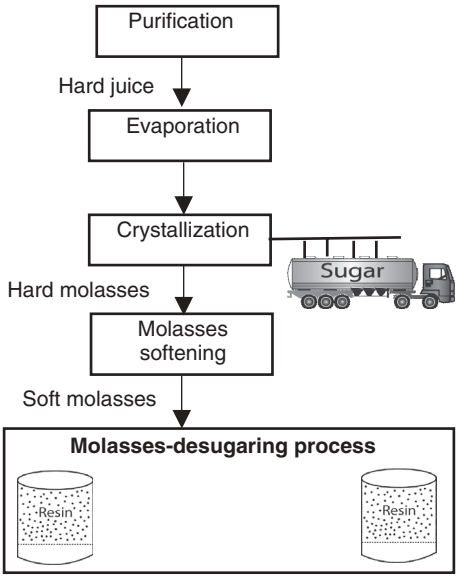


Figure 7.1 Flow diagram of molasses-softening process

Molasses-softening process is the decalcification of **hard molasses** (molasses with above 3.0 *mEq* CaO/100 *DS* hardness) by chemicals to produce **soft molasses**. Soft molasses is required for operations of the **molasses desugaring by chromatographic process** (see Chapter 8). As stated in the previous chapter, two approaches to molasses softening have been used in the beet-sugar industry:

- Thin-juice softening
- Molasses softening

The first method (discussed in Chapter 6) involves softening thin juice before concentration to thick juice and subsequent processing in the sugar-end into granulated-refined sugar and molasses. The molasses produced from the factory is free of **hardness** (soluble calcium and magnesium salts, also known as **limesalts**) because of its removal in the juice-softening process.

When the molasses to be fed to the separator is not soft (above 3.0 *mEq* CaO/100 *DS*), it must be chemically treated to reduce its hardness content to a safe level. Such molasses softening systems are similar to the water-softening process used to soften water for industrial use.

In the water-softening process, lime (CaO) and soda ash (Na_2CO_3) are added to the hard water. When lime is added to the water, it forms slaked lime:

$$CaO + H_2O \rightarrow Ca(OH)_2$$

Then the slaked lime reacts with Ca^{2+} in the hard water and the $(HCO_3)^-$ ion from the added soda ash to precipitate (↓) calcium carbonate:

$$Ca(OH)_2 + Ca^{2+} \text{ (from water)} + 2(HCO_3)^- \text{ (from soda ash)} \rightarrow 2CaCO_3\downarrow + 2H_2O$$

Therefore, for every mole of $Ca(OH)_2$ consumed, one mole of Ca^{2+} is removed from the hard water. In the molasses-softening process, unlike water softening, lime cannot be added to the

system because its reaction with the sucrose content of molasses creates saccharides. Instead, the caustic (NaOH) provides the hydroxyl ion required for this type of reaction. The chemistry of the molasses-softening process proceeds according to the following reaction:

$$2NaOH + Ca^{2+} \text{ (from molasses)} + 2(HCO_3)^- \text{ (from soda ash)} \rightarrow 2CaCO_3\downarrow + 2Na + 2H_2O$$

The optimum conditions for this reaction are

Temperature	50 to 80°C
Time	20 to 30 min
PH	10.0 to 10.5

The general procedure for molasses softening is straightforward. Theoretically, the chemistry of the process is the same for batch and continuous processes. Molasses is first diluted to approximately 60% dry substance (*DS*) and heated to approximately 50 to 80°C. (*The higher temperature is preferred for good filtration but increases color formation, which can be a problem for the separator.*)

An amount of soda ash equivalent to a 125 to 150% stoichiometric amount based on the total hardness of the molasses to be softened is mixed (dissolved) into the diluted and heated molasses. After the soda ash is dissolved, the pH of the mixture is adjusted to 10.0 to 10.5 with 50% caustic (NaOH) and allowed a reaction time with good agitation of 20 to 30 minutes, during which time the softening reaction will reach completion.

The pH can be adjusted before or simultaneously with soda ash addition to have the required hydroxyl ion (OH) in the media. The basic media is provided by the reaction of caustic with water, which is one of the most important factors affecting the substitution of Ca^{2+} and Na^+. Therefore, adding both chemicals *together* to the diluted molasses to achieve pH of 10.0 to 10.5 is recommended.

In general, the actual operating pH depends on the softening performance of the system. A higher pH is required if larger amounts of magnesium are present or if the molasses is generally more difficult to soften. During the reaction time, a sludge (precipitate) will form. The sludge is comprised mostly of calcium carbonate (the hardness in the molasses combined with the carbonate from the soda ash). This precipitated sludge is then separated from the molasses by filtration to complete the softening process. After the sludge is removed, the filtered molasses pH is adjusted downward with acid (hydrochloric or dilute sulfuric acid) to the range of 8.0 to 9.0 pH. This step prevents additional precipitation from the filtered molasses. (Note: Failure to adjust the pH could result in fouling the resin in the separator.)

MEASUREMENT OF HARDNESS IN MOLASSES

To properly operate the molasses-softening process, you must determine the hardness of the molasses to be treated. A number of laboratory methods can be used for such a determination, and the hardness may be expressed in a number of different units. For molasses, it is usually best to express its hardness either as milliequivalents (*mEq*) CaO or in equivalent (*Eq*) CaO and either on dry substance (100 *DS*) or on the sample. Such units make the calculation of the necessary amounts of chemical much easier. Typically, beet molasses have 5 to 40 *mEq* hardness/100 *DS*. Assuming a standard solids content of the molasses (80% *DS*), this would equate to 4 to 32 *mEq* hardness on the sample.

The hardness content of the molasses will vary, depending on soil conditions, beet quality, and the processing method for processing the sugarbeet into granulated-refined (GR) sugar. Molasses has a higher hardness where soils are strongly alkaline. If lime is used to control the pH of sugar-end syrups in the alkaline range, the molasses hardness will be higher. If soda ash is used in the beet-end to increase the alkalinity of the purified juice, hardness will be lower because the addition of soda ash will generally result in the precipitation of calcium carbonate ($CaCO_3$) in the purification process and lower the hardness in the thin juice.

ASSESSMENT OF PARAMETERS OF MOLASSES SOFTENING

No two molasses will respond the same to Molasses-softening and filtration operations. Due to the significant variation in the impurities in the molasses and the processing regimen to which the beets are subjected to and molasses produced, it is usually necessary to determine the parameters for chemical addition and process operation through laboratory scale testing of the molasses. Such tests are referred to as **jar tests**, and a series of such tests on the molasses to be softened will define the practical results of the softening process and the parameters to be followed for soda ash addition, pH control (with caustic), temperature, and reaction time. Usually, such testing will result in a table such as the following:

Starting molasses hardness	5 to 40 *mEq*/100 *DS*
Molasses concentration	Diluted to 60% from original concentration
Soda ash requirement	125 to 150% stoichiometric amount on molasses hardness
Caustic requirement	22 lb/t molasses
Reaction time	20 to 30 minutes depending on starting molasses hardness
Temperature	50 to 80°C (filtration rates favor higher temperature)
pH	10.0 to 10.5

After these parameters have been determined, the molasses-softening system can be properly designed and operated to produce the desired hardness of molasses. The preceding parameters are fairly typical of those required for a wide variety of beet molasses that might be subjected to chemical softening. Following are the amounts of expected solids to be produced based on the starting molasses hardness:

Hardness (*mEq*)	Solids (lb per ton molasses)
5	6.4
16	20.5
40	51.3

CONTINUOUS MOLASSES SOFTENING

The two types of molasses-softening systems are **batch** and **continuous**. Both systems include the following steps:

1. Molasses measurement
2. Molasses heating
3. Molasses dilution
4. Addition of chemicals (soda ash and caustic) to molasses

5. Sludge filtration
6. Adjustment of pH of the softened-filtered molasses
7. Final filtration

In addition to these primary steps, there is usually a need for a sweetwater system, filter aid systems for the filtration operation, surge tanks and air, and steam and water supplies for the process. Figure 7.2 shows a continuous-type molasses-softening system utilizing a high-efficiency reactor and a membrane filter press.

The advantages of a continuous-type system over a batch system are as follows:

- Lower installation costs
- Reduced color formation because lower residence times in the process
- The ability of the process to be tuned and operated from a central control room

In the system shown in Figure 7.2, the molasses is preheated, weighed in a batch scale, and then diluted with heated sweetwater to the desired concentration. After dilution, the molasses solution is heated to the reaction temperature. Soda ash is added as the molasses enters the three-stage reactor (see Figure 7.3). In the first compartment of the reactor, the molasses is thoroughly mixed with the soda ash. In the second reactor compartment, 50% caustic is added to increase the pH of the solution to the correct range, and mixing is continued.

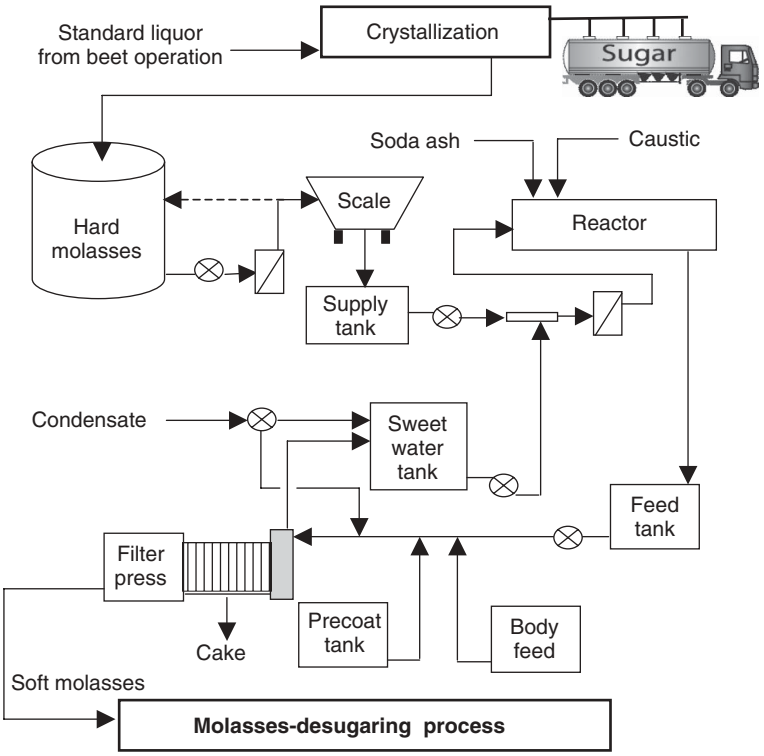


Figure 7.2 Detailed flow diagram of molasses-softening process

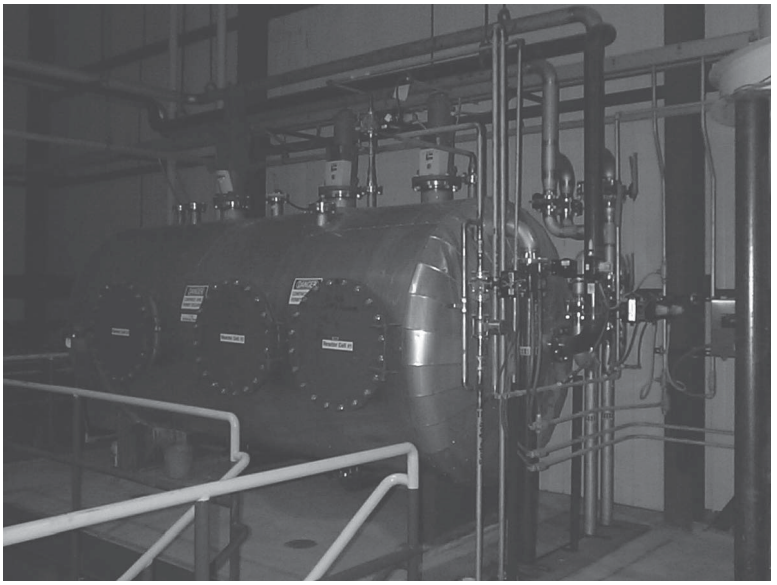


Figure 7.3 Molasses-softening reactor

In the third and final reactor compartment, agitation is maximized to drive the softening reactions to completion and maximize sludge production. The design of the system promotes relatively large sludge particles and ensures an easily filtered and dewatered sludge from the filter press. Usually, molasses filtration—especially the filtration of soft molasses produced from a factory employing thin-juice softening—is difficult. With a properly designed chemical softening system that produces large sludge particles, molasses filtration is much easier. Filtration rates for chemically softened molasses can easily be at least twice those of comparable thin-juice softened molasses.

The molasses-sludge mixture is sent to a supply tank that supplies the sludge-filtration section. In the filtration operation, a membrane filter press (see Figure 7.4) and a 100 t supply tank can handle a 350 t per day molasses-softening plant. Sufficient filtration capacity is required to allow for necessary cleaning and precoating of the filter press between its service cycles. It is recommended to have two filter presses, so one filter is always ready for service when the other one is off-line for cleaning or other reasons.

The sludge-free molasses is sent to the second supply tank from which the final molasses filtration is supplied on a continuous basis. The final filtration is performed to remove fine, suspended solids from soft molasses. Three pressure-leaf filters (Industrial filters), each with 300 m^2 (about 1000 ft^2) filtration area are used. One filter is used as polishing filter. Before entering the molasses-desugaring system, the pH of the soft-filtered molasses is adjusted downward from approximately 10.0 to about 8.8 to prevent any precipitation in the separators. Any such post-precipitation would adversely affect the operation and efficiency of the separators.

A **batch-type** system is similar to the system just described, except that the continuous reactor is replaced with a larger batch-type reactor in which molasses is mixed with water and chemicals in batches. A number of different batch configurations are possible, from a strictly batch operation to a semi-continuous operation having more than one batch reactor.



Figure 7.4 Membrane filter used for sludge filtration (Courtesy, H Putsch & Company)

Each system configuration has advantages and disadvantages. The major advantage of the batch system is that each completed batch of softened molasses can be tested before it is sent to the subsequent processing step. Some major disadvantages are increased labor requirements for the operation of the system and increased equipment costs and space requirements. Some major advantages of the continuous system are its low labor requirement and its ease of automation. Both types of systems are in operation, and both can give excellent results in terms of softening performance.

CALCULATION OF CHEMICAL REQUIREMENTS

If you know the original molasses hardness, it is easy to calculate the soda ash requirement for softening as well as the amount of sludge that will be produced. The amount of caustic required depends on the pH of the molasses and the amount of soda ash used. Higher magnesium levels require a higher pH than lower levels for complete removal of magnesium. Molasses having higher magnesium levels will also most likely give more problems with the final molasses filtration. For molasses normally low in magnesium content, the weight of 50% caustic on molasses is in the range of 0.2 to 1.0% on the weight of molasses processed. Filter aid consumption is a function of the type of filtration and the filterability of the molasses. A "normal" consumption of filter aid is between 1 kg (2.2 lb) per ton molasses and a high of 2 kg (4.4 lb) per ton molasses processed.

Soda ash consumption is a function of the original molasses hardness. In most cases, approximately 125 to 150% stoichiometric amounts of soda ash must be added relative to the CaO (hardness) content of the original molasses. For example, for a molasses with 40.0 *mEq* CaO/100 *DS* and the stoichiometric requirement of 125% soda ash on CaO (hardness) in molasses, the weight of soda ash required for softening a ton of molasses can be calculated as follows.

Stoichiometric means an amount relative to the **equivalent mass** (*Eq.*, see Section 1 in Chapter 12) of the reactants. In the case of softening, this would be the relative mass of the calcium being removed to the mass of sodium required to complete the reactions, and thus the

removal of the calcium or total hardness. When the reaction calls for a "stoichiometric requirement of 125%," this means the equivalent mass of sodium ion added for softening must be 125% of the equivalent mass of calcium ion present. Because the hardness in the molasses is measured as CaO, we must consider the molecular mass of the CaO (56) as the mass of what is to be removed and also the equivalent mass of the soda ash required to remove it.

The equivalent mass (*Eq*) of CaO is calculated using its molecular mass (*MM*) and number of charges (N_C) by the Eq. (12.5) given in Chapter 12:

$$Eq = \frac{MM}{N_C} = \frac{56}{2} = 28\,\text{g equivalent}$$

For the reactant soda ash (Na_2CO_3), the *MM* is 106 and the N_C is 2 because it contains two atoms of monovalent (+) sodium per molecule. Thus, the equivalent mass (*Eq*) is equal to half its molecular mass (106/2 = 53).

EXAMPLE

Given:

Molasses hardness	40 *mEq*/100 *DS*
Molasses dry substance (*DS*)	82.2%

Calculate:

- Equivalent CaO in one ton of hard molasses
- Amount of soda ash needed to achieve 125% stoichiometric addition rate per ton of hard molasses
- Soda ash used (% on molasses)

The amount of CaO if complete removal of hardness from the molasses is assumed, is

$$\frac{40}{100} \times \frac{82.0}{100} \times 1000\,\frac{\text{kg}}{\text{t}} \times 1000\,\frac{\text{g}}{\text{kg}} = 328000\,mEq\ \text{CaO/t}$$

$$328000 \times \frac{1\,\text{g}}{1000\,\text{mg}} = 328\ Eq\ \text{CaO/t molasses}$$

$$328 \times \frac{125}{100} \times \frac{106}{2} \times \frac{\text{kg}}{1000\,\text{g}} = 21.7\,\text{kg soda ash/t molasses}$$

$$\frac{21.7}{1000} \times 100 = 2.2\%\ \text{on molasses}$$

Using the equivalent-mass equation, and molecular mass of $CaCO_3$ (*MM* = 100) and that of CaO 56, the equivalent mass (*Eq*) of $CaCO_3$ is

$$Eq = \frac{MM}{N_C} = \frac{100}{2} = 50\,\text{g equivalent}$$

$$328 \times \frac{50}{1000} \times \frac{100}{56} = 29.3\,\text{kg}\ CaCO_3\text{/t molasses}$$

NOTE

- **It is much easier to do the basic calculations in metric units and convert the calculated data to the desired units of measure than to include the conversions within the initial calculations.**

PROCESS CONTROL

The **control** of the chemical-molasses softening system is straightforward. The main issues to deal with are ensuring that the correct temperature, pH, and addition of soda ash are maintained. Therefore, close observation and control of material flows and ratios are essential. Fully automated and properly alarmed processing variables in the processing systems go a long way toward obtaining the best results. Beyond these fundamental requirements, good reactor design having excellent mixing characteristics and adequate retention time will produce the desired results of a fully softened molasses and an easily filterable sludge.

Routine sampling of the product for the desired hardness level, pH and sediment ensures that the operation is in control and producing product within the specified control limits. Sampling must be frequent enough to match the tendency of the process to be maintained within control. Such sampling and analytical frequency will vary according to the system design and the functionality of the process as a stable system. Well-designed and stable systems can be operated solely from a remote control center, requiring only an attendant for process sampling and routine cleaning tasks associated with area sanitation and filtration operations.

OPERATING PROBLEMS

The main operating **problems** usually encountered are sediment in the softened molasses filtrate, which must be corrected within the filtration process, and high hardness, which must be corrected in the chemistry of the operation. The most likely causes of high hardness are improper temperature or pH control or inadequate addition of soda ash for the given hardness of the molasses being processed. In addition, mechanical issues with the processing equipment can cause a number of problems. Such problems must always be diagnosed and corrected when simple and appropriate adjustments of process variables do not solve a machinery malfunction.

END-OF-THE-CHAPTER NOTE

- **The molasses-softening process can be considered a bonus to the juice-softening process (the subject of the previous chapter) when a factory is equipped with the molasses desugaring by chromatographic (MDC) process (the subject of the next chapter). This is because not only can the factory desugar its own soft molasses but also it can desugar hard molasses purchased from the market.**

CHAPTER 8

MOLASSES-DESUGARING PROCESS

SECTIONS

- Molasses
- Molasses Desugaring by Steffen Process
- Molasses Desugaring by Chromatographic (MDC) Process
- Chemistry of the MDC Process
- Nonseparable Nonsucroses
- Conventional MDC Systems
- Modified MDC Systems
- Extract Processing
- Recovery of the MDC Process
- By-products of the MDC Process
- Installation of the MDC Process
- Economy of the MDC Process
- Cane-Molasses Desugaring
- Operating Problems

ABOUT THE CHAPTER **Chapter 8** discusses the most-talked-about subject in today's beet-sugar industry: the molasses-desugaring process. Conventional sugar-factory equipment cannot recover sugar left in molasses without using an additional process. That additional process is called the molasses-desugaring process. **Molasses desugaring by chromatographic** (MDC) **process** is the most advanced method for recovering up to 90% of the sugar present in molasses. It can increase the yield of sugar from beet from about 80% in a conventional beet factory to about 90%. The MDC process is an effective cost/return and environmentally saft technology, which greatly improves the efficiency of the factory and makes the beet-sugar industry more secure in the sweetener market.

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Sugar in molasses is valued lower (one-third to one-sixth) than granulated-refined (GR) sugar because of the low price of molasses. But the fact that molasses is valued less and contains about 50% sugar does not justify the installation of a desugaring project. Other important aspects, such as those discussed in this chapter, must be evaluated as well.

This chapter familiarizes you with the details of the MDC process (see Figure 8.1). The chapter focuses on technical information to help you select the best desugaring system. You also will be provided with the evaluation of the MDC process based on technical and economical factors to decide whether your factory is a good candidate for the installation of this technology.

Molasses desugaring has been the subject of study by sugar technologists since the beginning of the beet-sugar industry (the first beet-sugar factory was built in 1802 in Germany). **Molasses desugaring by chromatographic process** (from now on the **MDC process**) is the most advanced and efficient method for recovering sugar from molasses. Today, only a few suppliers offer MDC technology for application in the beet-sugar industry. The technology and equipment are complex and costly. The average capital cost of eight American MDC plants is about $35 M (almost one-tenth the cost of a complete beet-sugar factory). Therefore, detailed technical and economical information is required to help operating managers decide whether their facilities are suitable for the installation of the MDC process. Giving such information is the main objective of this chapter.

The MDC process is a new technology. It has been accepted since the late 1980s by beet-sugar companies, particularly in the United States, as an efficient way of increasing profitability. The first commercial MDC plant was built in Kantvik, Finland, in 1974. The first U.S. beet-sugar facility to install the MDC process was the Twin Falls factory in 1985. Since then, nine more beet factories in the United States have installed MDC technology. Two of these operations (the Fremont and Hereford factories) were shut down due to the installation of inappropriate technology. The remaining eight operations work successfully. Five of eight American beet-sugar companies are multi-member factories (each company has two to five factories) and all multi-members have equipped at least one of their factories with MDC technology since 1985. Each MDC plant has a processing capacity of 350 to 600 tons molasses per day and a total capacity of about 4500 tons per day among all processors. Today, over 90% of molasses produced by U.S. beet-sugar producers is desugared to increase the overall efficiency of the industry.

The three main economical reasons for using the MDC process by the beet-sugar industry are the following:

- Production costs of sugar produced from molasses is lower than that from beet
- The value of sugar in molasses is one-third to one-sixth that of granulated-refined sugar
- Recovering sugar from molasses benefits the factory more than growing more sugarbeet

In addition, a suitable MDC technology can make the beet-sugar industry more secure in the world sugar market and more competitive against the low-cost substitute sweetener industries

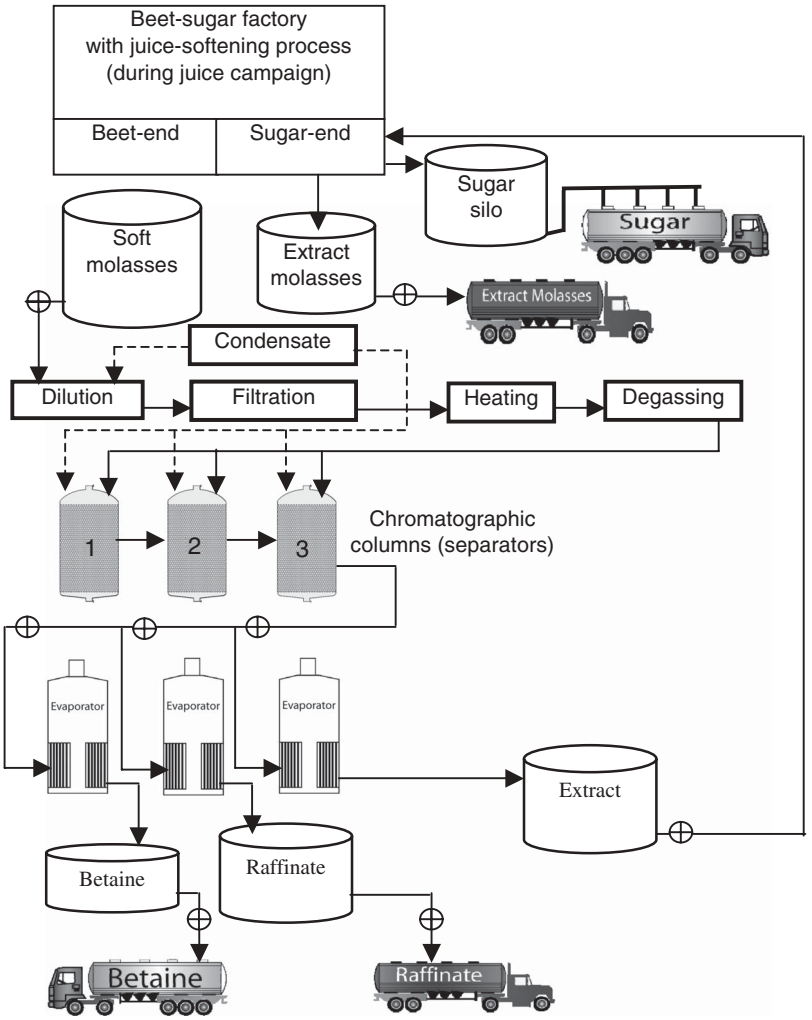


Figure 8.1 Beet-sugar factory with MDC process

(e.g., high-fructose corn syrup). It can also extend the equipment-utilization period of the factory and protect the factory when it does not have enough sugarbeet.

Technologically, a suitable MDC process can achieve the following:

- Sugar recovery through separation (90+%)
- Sugar recovery through crystallization (78+%)
- An extract fraction with low color and high purity (90+%)
- Increase the sugar yield (extraction) of the factory by about 10%

NOTES

- **Whether or not a beet-sugar factory is equipped with the MDC process, production of molasses with high purity is the worst mistake that any sugar factory can make. The molasses produced from the beet juice or extract (the product of the MDC process) must have the lowest possible purity, called satisfactory molasses (the molasses with a purity of 60% or below). In other words, the factory must process beets effectively to produce:**
 - **Sugar with the highest possible recovery**
 - **Molasses with the lowest possible purity**
- **Beet-sugar technologists should recognize the importance of this objective, since at equal amounts of beet processed, every ton of extra sugar lost to molasses decreases sugar production, increases the production costs of sugar from beet, and produces extra molasses. Desugaring of extra molasses in the MDC process (if the factory is equipped with this process) increases the production costs of sugar from molasses. As a result, the total production cost (from beet and molasses) is increased, causing considerable reduction in profitability of the factory.**

MOLASSES

Molasses is defined as the **mother liquor** (runoff syrup) from the final stage of crystallization. The eight most important properties of beet molasses are the following:

- Is produced at about 5% on beet
- Has a high concentration (about 80%)
- Has a high amount of sugar (about 50%)
- Has about 15% of sugar coming with the beets
- Has about 80% of sugar loss of the beet-sugar factory
- Has as its most valuable components sugar, betaine, and amino acids
- Can be desugared by the MDC process to recover sugar, betaine, and minerals
- Can be used in animal feed, yeast, citric acid, alcohol, and pharmaceutical industries

Sugarbeet contains **sugar** (sucrose) and **nonsugars** (nonsucroses) such as betaine, amino acids, invert sugar, raffinose, and other water-soluble substances. In the diffusion station, sugar and nonsugars move from sliced beets (cossettes) to the juice, called the **diffusion juice**. In the purification station, some of the nonsugars are removed from the juice and some move through the process along with the sugar. In the crystallization station, the major part of the sugar (about 80%) is separated from the nonsugars and ends up in the sugar silo as the product of the factory. The rest of the sugar and all nonsugars end up in molasses. Figure 8.2 shows how molasses is produced (formed) in a beet-sugar factory (see also Section 17 of Chapter 3).

Soft molasses (raw material of the MDC process), simple speaking, is almost free of **hardness** (limesalts, soluble salts of Ca and Mg). Chemically speaking, its hardness content equal to, or less than, 3 *mEq*/100 *DS* (reads milliequivalent per 100 grams of dry substance) as CaO (discussed in Chapter 6). This equates to 85 mg/100 *DS* (reads milligrams per 100 of dry substance) or 850 ppm. Equivalent mass formula (given in Chapter 12) can convert the hardness units. The equivalent mass (*Eq*) is based on the molecular mass (*MM*) of the substance and its number of charges (N_C):

$$Eq_{CaO} = \frac{MM}{N_C} = \frac{56}{2} = 28 \qquad \frac{85}{28} = 3\,\text{milliequivalent}\,(mEq)$$

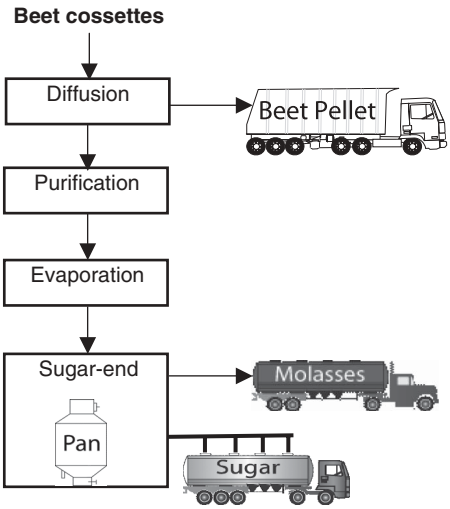


Figure 8.2 Molasses formation

Hard molasses has a hardness (limesalts) content higher than 3 *mEq*. Depending on thin-juice hardness content, typically beet molasses have 5 to 40 *mEq*/100 *DS*. Hard molasses has many undesirable qualities for the MDC process. More importantly, when hard molasses is fed into the **separator** (chromatographic column filled with resin), the calcium (Ca) and magnesium (Mg) cations bond with the resin and block its pores. This decreases the separation capacity of the resin, resulting in lower performance of the separator and, consequently, shuts down the MDC operation.

In the previous two chapters, you learned about two methods for the preparation of soft molasses:

- Thin-juice softening
- Molasses softening

Figure 8.3 shows a simplified flow diagram of two beet-sugar factories with the MDC process: one with thin-juice softening and the other with molasses softening. Note: Some U.S. factories are equipped with both softening technologies. This puts their MDC process into a two-mode operation:

- To desugar their own soft molasses
- To soften hard molasses purchased from the market and then to desugar it

MOLASSES DESUGARING BY STEFFEN PROCESS

Molasses desugaring by Steffen process is the oldest desugaring method. It was patented by Carl Steffen in 1883. The Steffen process can desugar molasses with any amount of hardness content, which is considered as its advantage over the MDC process. Although it is still in use in some beet-sugar factories, the Steffen process is no longer popular because of the following disadvantages:

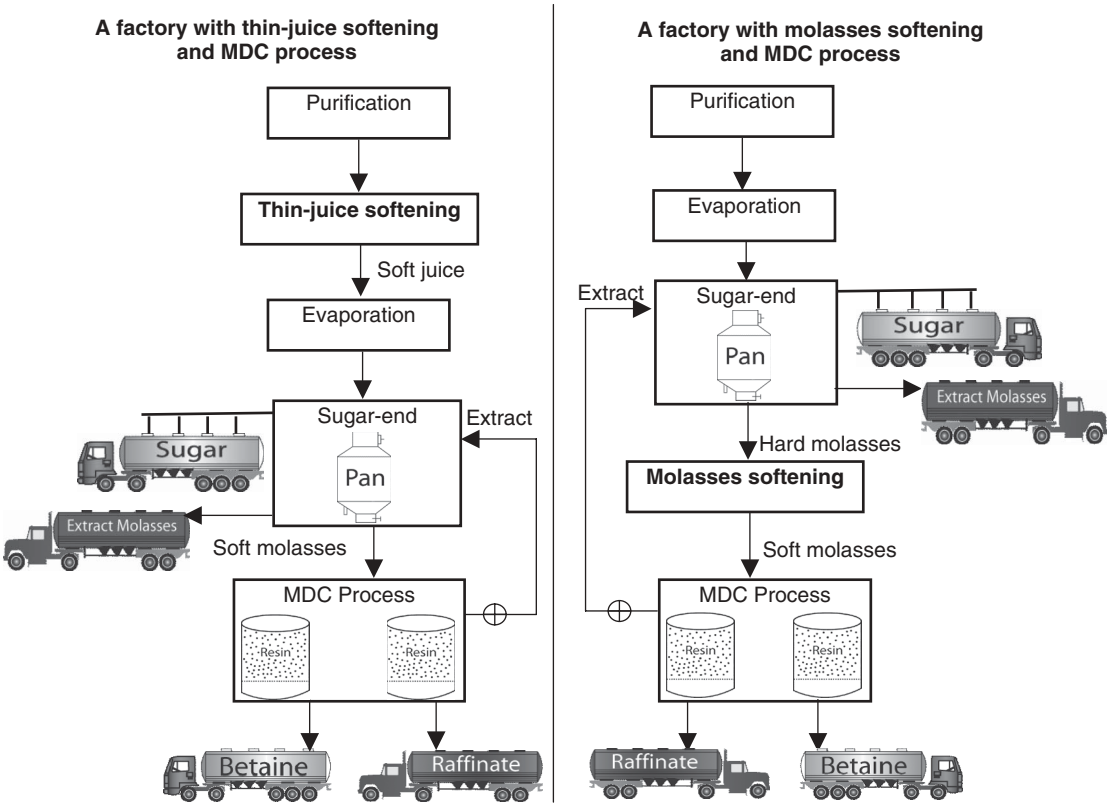


Figure 8.3 Ways of producing soft molasses

- A single-component process (separates only sucrose)
- Low sucrose recovery (about 60% of sucrose in molasses)
- High amount of wastewater production (about 800% on molasses processed)
- Difficulty in operating the cooling system and the fine quicklime-production process

Note: The Steffen process can recover up to 85% of the sugar in molasses, but it carries about 15% of nonsugars back to the purification station. The recycle nonsugars can carry about 25% of sugar to molasses (based on the rule of thumb used in the beet-sugar industry: each kg of nonsugar carries about 1.5 kg of sugar to molasses). This results in an actual sugar recovery of about 60%.

The Steffen process is based on the following principles:

- Precipitation (↓) of sucrose by CaO to tri-calcium saccharate

$$C_{12}H_{22}O_{11} + 3CaO \rightarrow C_{12}H_{22}O_{11}.3CaO\downarrow$$

- Low solubility of tri-calcium saccharate in water (even at higher temperatures)

The following are the highlights of the operation of the Steffen process:

1. Molasses is diluted to 10% *DS* using wash water from saccharate filters.
2. The diluted molasses is vigorously agitated while fine powder of calcium oxide (CaO, quicklime) is added to it. The mixture is cooled by cooling water to about 10°C.
3. The saccharate precipitate is separated by filtration, while nonsugars stay in solution.
4. The filtrate is heated to 90°C to precipitate more saccharate. The tri-calcium saccharate has very low solubility even at high temperatures, which is helpful in the precipitation of more sucrose in the form of saccharate when the filtrate is heated.
5. The precipitate is filtered again to produce the second filter cake.
6. Both filter-cakes are mixed with sweetwater (from the filtration process) to produce saccharate milk, which is used in the purification station along with fresh milk of lime.

NOTE

Readers interested in more information on the Steffen process can refer to *Beet-Sugar Technology* (McGinnis 1982) page 595.

MOLASSES DESUGARING BY CHROMATOGRAPHIC (MDC) PROCESS

Molasses desugaring by chromatographic process (MDC process) is the newest method for recovering sugar left in molasses. The MDC process is based on **ion-exclusion chromatography** that was invented in the United States in the 1950s (Wheaton et al. 1953). Ion-exclusion chromatography was first used commercially by the U.S. corn industry to produce high-fructose corn syrup (HFCS). The process then attracted the attention of beet-sugar researchers who started to develop it to recover sugar from molasses. Hongisto (1976), Schoenrock (1983), and a few other sugar technologists were among the first to conduct research that led to the development of the MDC process. Later, a few more scientists modified the existing technology to the extent that today MDC is taken by the beet-sugar industry as the most effective way to secure the industry in the world sugar market.

The MDC process has the following advantages compared to the Steffen process:

- MDC is a multiplecomponent process (separates more components present in molasses)
- MDC is a high-efficient process (recovers about 90% of sucrose in molasses)
- MDC is a wastewater-free process

The MDC process is a resin-based ion-exclusion chromatography that works based on the **exclusion** (rejection) of **ionic compounds** (which decompose to positive and negative ions when dissolved in water, e.g., most nonsucroses) and the **inclusion** (absorption) of **nonionic compounds** (which do not decompose to ions when dissolved in water, e.g., sucrose). Compared to other types of chromatographies, **ion-exclusion chromotography** (IEC) offers the following advantages:

- IEC does not need resin regeneration
- IEC can handle higher feed concentration
- IEC does not replace cations for anions, and vice versa

The MDC process is a multicomponent process separating not only the sucrose fraction (**extract**) from nonsucrose fraction (**rafinate**) but also recovering other valuable components

of molasses such as **betaine** (the by-product of the MDC process with about 50% betaine purity).

The MDC process requires considerable capital costs and a large area for equipment installation to the extent that it is considered a plant. The MDC plant (chromatographic columns and auxiliary equipment such as evaporators for concentrating byproducts, holding tanks, and filters) is a stand-alone operation and is usually installed next to the main operating building of the factory. MDC equipment is usually installed in a separate building that is connected to the main beet-processing building of the factory to use steam, power, and utility systems.

Before we get to the chemistry of the MDC process, it is helpful to briefly outline the steps involved in this process:

1. **Dilution:** Soft molasses is diluted to 60 to 70% *DS* with soft water (usually condensate).
2. **Filtration:** Diluted molasses is filtered to remove suspended solids to prevent resin fouling.
3. **Heating:** Filtered juice is heated.
4. **Degassing:** Heated juice is deaerated to prevent pressure fluctuations in the separator.
5. **Feeding:** A volume of feed (less than the volume of resin) is fed through a proper distributor to the separator containing packed resin in sodium (Na) or potassium (K) form.
6. **Elution:** After the feed has passed down the separator, flow continues by soft and degassed water in a water-to-feed ratio of about 6 to 1.

As the feed passes through the resin, the nonionic substances (mainly sucrose) attach to the resin, and ionic substances (mainly nonsucroses) continue moving through the resin bed. The ionic-compounds fraction generally known as **raffinate** (in the sugar industry, CMS for concentrated molasses solids), is collected first. Then the elution water elutes (removes) sucrose and other nonionic-components from the resin. This fraction is collected separately and called **extract**.

Extract (the high-sugar fraction) is the product of the MDC process and is high in sucrose (90+% purity) and low in nonsucroses (below 10% impurities on *DS*). It is usually concentrated to about 70% dry substance (*DS*) in the extract evaporator and stored in the extract storage tank. Later, it is crystallized into granulated-refined (GR) sugar during the extract (juice) campaign. (Note: More will come later in the Extract Processing section in this chapter on the ways that extract is processed.)

CMS (the low-sugar fraction) is the byproduct of the MDC process and is low in sucrose (about 15% purity) and high in nonsucroses (about 85% impurities on *DS*). Raffinate has a *DS* of about 12%. It is concentrated to about 60% and stored in a tank. CMS is usually sold to the animal feed industry but has a number of other industrial uses as well.

In the MDC process, extract purity and raffinate *DS* can be adjusted by changing the following factors:

- Step time
- Flow rate
- Amount of elution water
- Output ratio of extract to raffinate

Today, the three-fraction operation is the most common type of the MDC operation because it can separate betaine in addition to the other two fractions (extract and CMS). The amount of **betaine** (a nitrogenous compound) in beet molasses is high (about 5% of the solids in molas-

ses). The three-fraction operation can recover a considerable amount of betaine (90+%) in the form of about 50% purity. The betaine fraction is concentrated (from about 12 to 60%) and sold as **concentrated-liquid betaine** (CLB). It is used mainly in the animal feed industry, although it has other uses as well.

NOTE

For CMS and betaine qualities, see the By-products section of this chapter and also Chapter 4. For analytical information on these by-products, refer to Section 8 of Chapter 11.

CHEMISTRY OF THE MDC PROCESS

In molasses desugaring by chromatographic process (MDC process), the sucrose in molasses separates from the nonsucroses based on the following principles:

- Different affinities (absorption capabilities) for ionic and nonionic components
- Exclusion (rejection) by resins of ionic components and inclusion (adsorption) of nonionic components
- Differing molecular masses of components in molasses

When ionic substances (e.g., salt) are dissolved in water, their elements separate to form **ions** (cations with positive charges and anions with negative charges). Unlike ionic substances, when sucrose (a nonionic substance) is dissolved in water, its elements (carbon, hydrogen, and oxygen) do not separate; its molecules stay intact and combine with water molecules to form a **hydrated-sucrose solution** ($C_{12}H_{22}O_{11}.H_2O$). The molecules held in a group of water molecules are said to be in a **solvent cage**. The number of water molecules in the sucrose-water solvent cage varies, depending on the concentration of the solution (see also Section 4 of Chapter 1).

Let us see what happens when diluted molasses is fed into a column which is filled with a particular resin (styrene-divinylbenzen-based resin with an average particle size of about 0.3 mm) and followed by passing water (water-to-feed ratio of about 6 to 1) through the resin. The nonsucroses (ionic) are **excluded** (rejected) from the resin, moving through quickly while the sucrose molecules (nonionic) are **adsorbed** (see the following Note) by the resin, thus moving through more slowly. At the same time, water reaches the resin, penetrates to its pores, dissolves the sucrose, and removes it from the resin. As a result, sucrose appears later in the **effluent** (output solution from the column). Chemically, a series of **rejection-absorption** actions between the resin and the components in molasses take place, causing sucrose molecules to stay behind. As in a race, a competition between faster components (nonsucroses) and the slower one (sucrose) occurs. The winners are nonsucroses, which reach the end of the column and appear in the effluent first while sucrose appears later (after eluting the resin with water).

NOTE

Specialists on chromatography use the word *adsorption* and *absorption* when they talk about ion-exclusion chromatography. The term adsorption comes from *adsorb* (to adhere) meaning to stick to the surface of a substance, like some nonsucroses stick to the surface of calcium carbonate during juice purification. The term absorption comes from *absorb* (to attract), meaning to penetrate into the interior of something, as when water penetrates into a sponge. In ion-exclusion chromatography; both actions occur because the separation of components occurs when simultaneously small molecules of nonionic components penetrate (absorb) into the resin pores and larger molecules stick (adhere) to the surface of the resin.

Because of the Donnan membrane effect, the nonionic components in the ion-exclusion process have a tendency to stay in equal concentration in both the resin phase and the liquid phase, while the ionic components stay at much higher concentration in the liquid phase than in the resin phase. Therefore, during the separation, the ionic components move much faster than the nonionic. In other words, the separation takes place because the **solutes** (a general name for components being dissolved, in our case, sucrose and nonsucroses) have different distribution coefficients. The **distribution coefficient** (D) determines mathematically the affinity of the resin for a particular component. The **mass distribution coefficient** (D_{MASS}) for component A is given as

$$D_{MASS} = \frac{[A]_{resin}}{[A]_{solution}} \tag{8.1}$$

In this equation, the brackets indicate the concentration. The difference in the distribution coefficient is a measure of the separability of the solutes (*the larger the D value, the greater is the affinity of the component for resin and, consequently, the slower the component travels through the resin bed*). For example, sucrose has a larger distribution coefficient ($D = 6$) than most salts, so it has a greater affinity for the resin, causing a slower movement than salts. The separation of the main components of molasses occurs in the following order (from faster to slower):

- Salts
- Sucrose
- Invert Sugar
- Betaine

Note: When two components have about the same affinity, the component with the higher molecular mass (*MM*) travels faster through the resin than the component with the lower *MM*. For example, sucrose has a higher *MM* (342 g) than invert sugar (180 g), so it travels faster than invert sugar and appears earlier in the effluent. This happens because large molecules cannot penetrate into the pores of the resin as easily as the small molecules. In the same way, part of colorants (with high *MM*) is removed in the separator because of considerable *MM* difference of colorant and sucrose. So, resin pore size is important (*the higher the DVB content, the larger is the pore size*). DVB stands for divinylbenzen (see Chapter 5).

In general, the rate of separation of sucrose from nonsucroses depends on the following:

- Feed flow rate
- Feed temperature
- Feed concentration
- Feed hardness content
- System design and the control system
- Amount of elution water (water-feed ratio)
- Feed distribution system at the top of the separator
- Product collection system at the bottom of the separator
- Resin properties (particle size, uniformity, and DVB content)
- Height of the resin bed (*the longer the bed, the greater is the separation*)

Producing extract with high purity along with high sugar recovery are the main objectives of the MDC process. To improve these values, the degree of separability of the difficult-to-separate components (**nonseparable nonsucroses**) of molasses must be improved, as explained next.

NONSEPARABLE NONSUCROSES

Nonseparable nonsucroses (NSNS, see the following Notes) are the difficult-to-separate components of molasses during the MDC operation. This is because, during separation, some nonsucroses (e.g., invert sugar, betaine, and amino acids) move closely with the sucrose and end up in the extract fraction. NSNS affects separation efficiency considerably (see the Notes below).

The NSNS are difficult to separate from sucrose for one or more of the following reasons:

- Rate of movement between sucrose and NSNS is similar
- Affinity of the resin for sucrose and NSNS is similar
- Molecular mass of sucrose and NSNS is similar

The degree of separability of NSNS is the most important subject in MDC technology with different views with respect to their separability improvement. Some chromatographic scientists suggest that the conditions of the separation in the first stage of separation must be kept in such a way as to force sucrose to behave like an excluded component, and thus to move with the nonsucrose components and end up in the sucrose-salt fraction (raffinate). This allows the efficient separation of components with low molecular mass (e.g., invert sugar). Then, the sucrose-salts fraction is treated in another column in which the sucrose is separated from the salts (Kearney 1997).

NOTES

- **Some sugar technologists refer to nonseparable nonsucroses (NSNS) as extract contaminants because they mix with the extract fraction and decrease its purity. Some call them overlapping components, separator nonremovable nonsucroses, or crossover nonsucroses. The term nonseparable nonsucroses (NSNS) is used in this book.**
- **In the sugar industry, the term *n*on*r*emovable *nonsucroses* (NRNS) is usually used in the juice-purification process to refer to nonremovable nonsucroses when the juice is limed (see Section 10 of Chapter 3). There is a difference between NRNS and NSNS.**
- **Nonseparable nonsucroses affect not only separation efficiency during the desugaring of molasses but also crystallization rate during the crystallization of extract (*the higher the NSNS content of extract, the lower is the crystallization rate*).**
- **During the crystallization of extract, the NSNS accumulate in the extract molasses (secondary molasses). If the extract molasses is reprocessed in the separator, the NSNS start to accumulate and reduce the performance of the separator (see the Processing Extract section of this chapter). Therefore, the extract molasses must be kept separately from virgin molasses (molasses that has not gone through the separator).**
- **Extract molasses has a similar purity as virgin molasses (about 60%), but it cannot be desugared again because it is high in NSNS.**
- **Chromatographic specialists have been working on the separability of the NSNS to improve the efficiency of the MDC process. This led them to invent continuous and later modified MDC systems, described next.**

CONVENTIONAL MDC SYSTEMS

Unlike batch chromatography (shown in Figure 8.4), which operates in a fixed-bed way, the continuous systems are moving-bed chromatography (shown in Figure 8.5). Continuous chromatography provides higher efficiency than the batch systems because its mass-transfer zone is over the whole part of the bed height. In other words, the molasses solution is actively in contact with the resin during the separating process. The disadvantages of the batch system, as compared with the continuous system, are the following:

- Inefficient resin utilization
- High elution-water requirement
- Inefficient in three-component separation

The application of **true** continuous chromatography is problematic because when the resin is pumped continuously from the bottom of the column to the top or from one column to the next, the resin quickly becomes damaged, reducing its life expectancy. Therefore, this system is not used in the industry. To overcome the shortage of resin movement, chromatographic researchers have invented two improved methods:

- Continuous simulated moving bed
- Continuous sequential moving bed

Continuous Simulated Moving Bed

Continuous simulated moving bed (continuous SMB) is a chromatographic system in which the resin remains fixed and the inputs and outputs move around the resin. This system is called

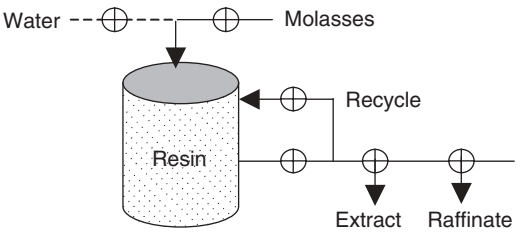


Figure 8.4 Batch process

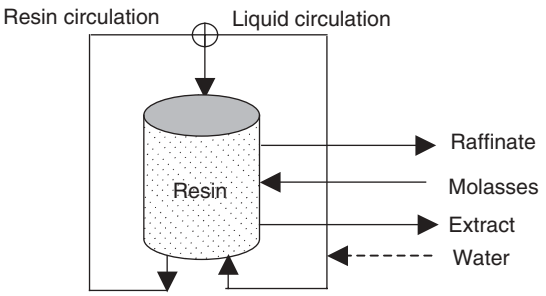


Figure 8.5 Continuous moving-bed process

simulated moving bed because the resin itself does not move, but rather, the flows simulate the movement of the resin. In other words, the inputs (molasses and elution water) to the column and the outputs (extract and raffinate) from the column are moved to the next column. The movement does not interrupt the input and output flows.

In continuous SMB, the feed circulates continuously in a closed loop. No beginning or end exists because at a regular interval (called the **step time**), the position of each input and output simultaneously changes to the next column. Circulating pumps installed between each column maintain a steady flow through all the columns (usually three to eight). The resin remains in a fixed position but acts against the liquid flow. As a result, the components are separated over the resin, resulting in better separation of NSNS.

In a typical continuous SMB system, the feed and elution water are continuously added to the columns, while extract and raffinate fractions are continuously withdrawn from the columns. For example, as shown in Figure 8.6, in an eight-column system with two fractions (extract and raffinate), elution water is added to column 1, extract is withdrawn from column 3, feed is added to column 5, and raffinate is withdrawn from column 7 simultaneously. Then, at fixed intervals, the elution water is added to column 2, the extract is withdrawn from column 4, feed is added to column 6, and raffinate is withdrawn from column 8.

Sequential Simulated Moving Bed

Sequential simulated moving bed (sequential SMB) is a chromatographic system in which the inputs and outputs operate sequentially, meaning that the flows are interrupted during the operational cycle at regular intervals. Inputs (feed and water) are added to different points in the system, and outputs (raffinate and extract) are collected discontinuously at fixed points from the system.

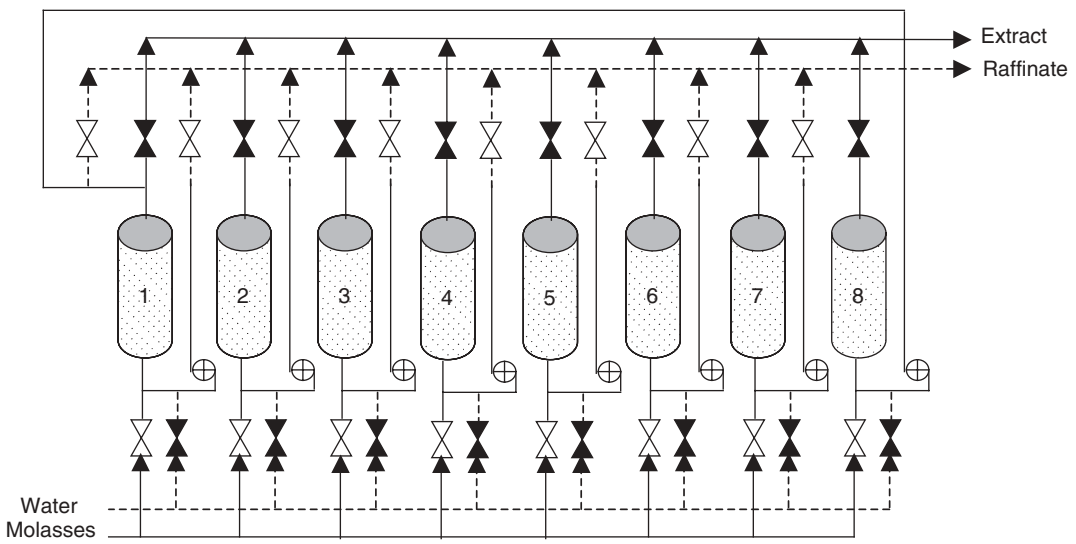


Figure 8.6 Continuous SMB process with two fractions (extract and raffinate)

Despite the fact that in **sequential SMB**, the input and output flows are sequentially interrupted, the system is considered a continuous process because the concentration difference within the column moves constantly as the flows are changed to the next column.

A special rotary-valve system is used to introduce the feed and elution water and to draw outputs. A typical sequential process has three columns with a resin-bed height of about 5 m. The system can efficiently operate in a three-fraction separation mode, producing extract, raffinate, and betaine (Paananen 1997).

MODIFIED MDC SYSTEMS

Because conventional SMB systems were not able to operate efficiently in multi-fraction separation (particularly in the area of separating betaine), sugar scientists continued to modify SMB systems. Both conventional and modified systems operate continuously, but there are some differences between the systems as follows:

- Method of treating NSNS components
- Interconnection of the columns
- Feed distribution design
- Height of the resin bed
- Feeding system design
- Construction details
- Number of columns
- Control methods

Before the 1990s, the five major suppliers of the MDC process were

- Applexion system (offered by Applexion, France)
- ARI system (offered by Amalgamated Research Inc., United States)
- Finn Sugar system (offered by Finn Sugar, Finland)
- IWT system (offered by Illinois Water Treatment, United States)
- Mitsubishi system (Japan)

Due to joint ventures among MDC suppliers, presently there are only two major suppliers:

- ARI system (Amalgamated Research Inc.)
- FAST system (Finnsugar-Applexion Separation Technology)

ARI System

ARI offered its modified system in 1997 under the name **coupled-loop chromatography** (CLC). This system was installed in the Twin Falls, East Grand Forks, Nampa, Monitor, and Hillsboro factories in the United States.

The CLC system (see Figure 8.7) consists of two chromatographic sections, called **loops**, that work independently. The soft-diluted molasses (about 60% *DS*) and elution water are fed to loop 1, which produces an **upgrade fraction** and a betaine fraction. The upgrade (with about 35% *DS* and 65% purity) goes through the evaporation process to be concentrated to about

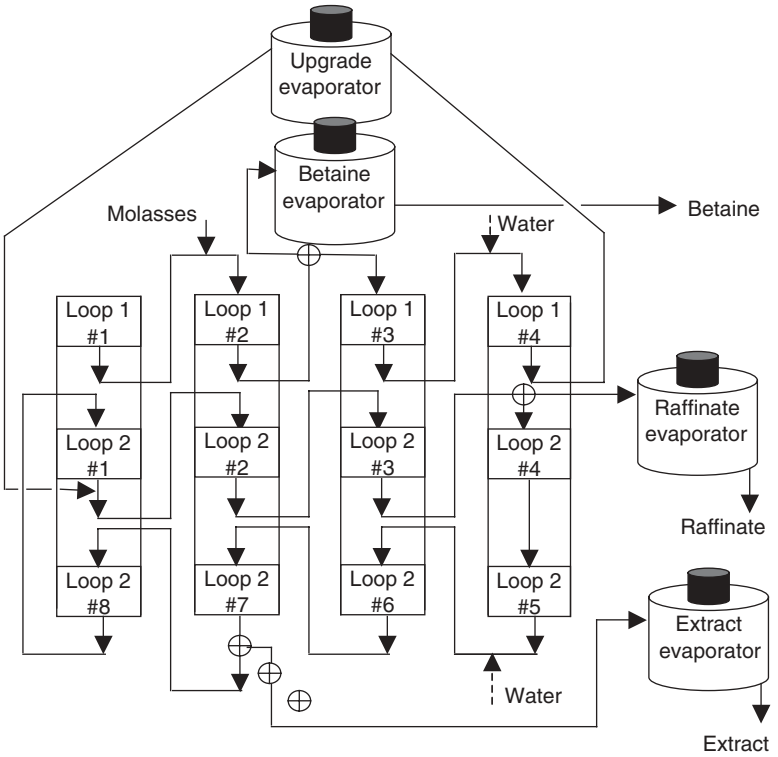


Figure 8.7 Flow diagram of coupled-loop MDC process

70%. Then, it is fed to loop 2, which produces extract with 92+% purity and raffinate with about 12% purity.

Typically, loop 1 consists of four cells and loop 2 has eight cells. Usually, four cells are stacked to occupy less floor space and provide an easier interconnection between the cells and loops. The stack of four cells and accessories between the cells make a column about 5 stories (about 20 m or 70 ft) high. The resin bed in each cell is about 3 m high and 3 to 6 m in diameter. Each cell contains 15 to 30 m^3 (530 to 1060 ft^3) resin. Figure 8.8 shows the chromatographic columns and their interconnecting pipes and parts.

The main advantage of the CLC system is in the treatment of nonseparable nonsucroses (NSNS) during separation. In a conventional SMB system, sucrose behaves as a slower acting component relative to the salts and components with a high molecular mass. As mentioned previously, the invert sugar and other components with a low molecular mass move close to sucrose, making them difficult to separate from the sucrose fraction. In contrast, in the CLC system, the conditions of loop 1 force sucrose to behave as an excluded component and move with the salts fraction. Then, the output from loop 1 (upgrade) enters loop 2, which works independently from loop 1. Here, the sucrose separates from the salts and components with a low-molecular mass. This improves the separability of the NSNS components.

Evaporation of the upgrade fraction between the two loops is considered an advantage because a feed with high *DS* results in a higher sucrose recovery. Another advantage of the



Figure 8.8 Chromatographic columns (Courtesy, Michigan Sugar Company)

CLC system is that the sucrose can be held in the separator for a longer period. This increases the contact time of sucrose with the resin, improving sucrose separability. As a result, the following improvements are achieved:

- Higher extract *DS*
- Higher extract purity
- Higher sucrose recovery
- Higher color elimination

In a pilot study (Kearny 1997), at a water-to-feed ratio of 5.6 to 5.8, the CLC system had the following results:

Feed purity (%)	60
Extract purity (%)	97
Extract *DS* (%)	41
Extract color (IU)	4500
Raffinate purity (%)	17
Betaine recovery (%)	95

Fractal technology (see Figure 8.9) is a method for distributing liquid in the MDC process and the thin-juice softening process. The fractal distributor has been used since 1999 in ARI chromatographic columns. This type of distributor operates efficiently because resins receive their equal share of feed. The result is a uniform and efficient feed distribution, improving the performance of the systems (Kearney et al. 1999).

FAST System

Finnsugar Applexion separation technology (FAST) is a system developed by a joint venture between Finnsugar and Applexion. The FAST system has been on the market since the late

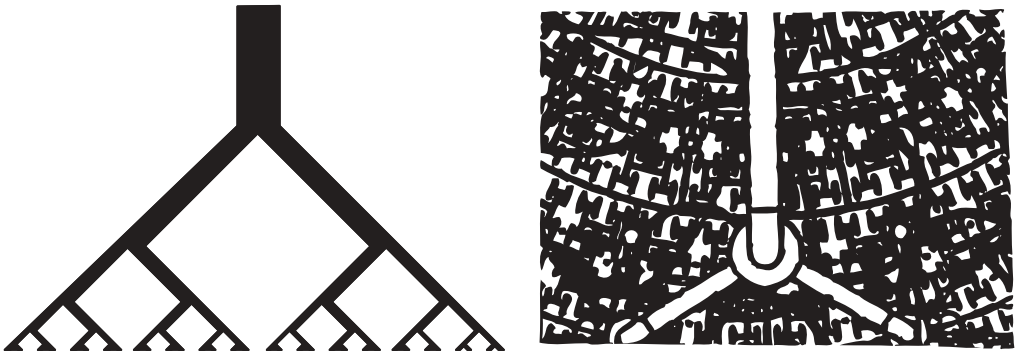


Figure 8.9 Fractal distribution system

1990s. This system offers the latest improvements in desugaring technology. This is a modified version of the sequential SMB that offers effective separation of the NSNS components. This system was installed in the early 1990s in the Renville and Scotts Bluff factories in the United States and in several other factories around the world, with satisfactory operations.

The operation of the FAST system (see Figure 8.10) is simple and is based on the three-column sequential process with a resin bed height of about 5 m. It uses three columns, and molasses is fed to one column (called the feeding column) at specific intervals (Paananen 1997 and 2000). For example, assume that molasses is fed into the first (feeding) column, then the process continues as follows:

1. When the **salt fraction** (raffinate) reaches the bottom of the first column, the raffinate valve opens to remove the raffinate until the sucrose fraction reaches a certain level. At the same time, the sucrose fraction is removed from the third column, and elution water enters the second column.
2. After the feed has entered the first column, the concentration difference in the system is circulated. The betaine fraction is taken out of the third column by adding water to the first column.
3. The circulation process continues until the raffinate fraction reaches the bottom of the second column. At this point, raffinate is taken out of the second column, and water is introduced into the third column.
4. Circulation continues until the raffinate reaches the bottom of the third column. The raffinate is taken out by introducing water into the first column.
5. When the sucrose content at the bottom of the third column increases to a certain level, the concentration difference is circulated again.

By repeating these steps, the separation of molasses into multiple components is achieved.

NOTE

The Illinois Water Treatment (IWT) process, called the ADSEP process, was used in the late 1980s in the Fremont factory (in Ohio) to process 400 tons molasses per day. Before that, the ADSEP technology was used in glucose-fructose separation. The ADSEP process used sequential SMB. The Fremont operation was shut down in the mid-1990s after a few years of inefficient operation.

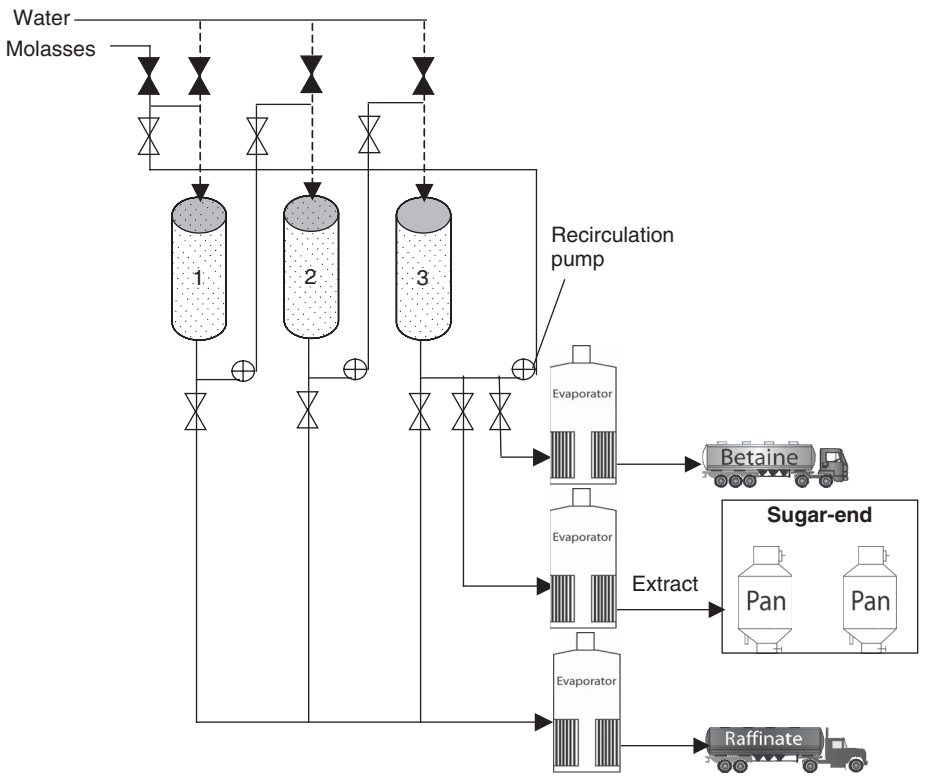


Figure 8.10 Flow diagram of FAST MDC process

EXTRACT PROCESSING

Extract (the high-sucrose fraction) is the product of the MDC process and is processed in the sugar-end into GR sugar (granulated-refined sugar) and secondary molasses (extract molasses). The amount of extract molasses produced is small, typically about 10 to 12% on the mass of molasses desugared or about 0.5 to 0.6% on beet processed (if all the molasses from the beet crop is desugared). Extract processing is a new chapter in sugar technology, so little information has been published about it. This section provides an overview.

The extract is normally processed into sugar in the following ways:

- Separate crystallization during the extract campaign
- Cocrystallization during the beet campaign
- Copurification during the beet campaign

The **separate crystallization** refers to a stand-alone processing of extract during the extract campaign (during the off-beet campaign). This method is efficient because it isolates the non-separable nonsucroses (NSNS) for the extract molasses. The separate accumulation of NSNS prevents their accumulation in the sugar-end, resulting in a higher sugar recovery. But the sugar-end operation may face high-color profile, resulting in a high-color sugar production.

By this method, the concentrated extract is stored in the tank until the end of the beet campaign and then processed separately in the sugar-end during the extract campaign. During this off-beet period, only the sugar-end, boilers, and evaporators operate to process the stored extract.

If the extract has high purity (94+%), the sugar-end may experience a high-purity profile all the way to molasses. This can be solved by the following precautions:

- Lowering the purity of the high-raw (B) crystallization to a reasonable 86 to 88% by back-boiling (see recommendation given in Section 17 of Chapter 3)
- Adding a crystallization stage (from three to four stages)
- Conducting crystallization at a higher supersaturation (about 1.22)
- Maintaining the operation of the separator ina way to produce extract with no purity above 94%.

Cocrystallization of the extract during the beet campaign has been tried in some factories, where the sugar-end capacity is larger than the beet-end throughput. By this method, the extract is mixed with standard liquor from beet processing, and the process continues in a manner similar to conventional crystallization.

An important issue is involved with the cocrystallization method: The ratio of extract to standard liquor should be substantially lowered for the reasons mentioned next. At any rate of cocrystallization, a steady buildup of the NSNS occurs. This results in a considerable drop in extract purity and sucrose recovery in the MDC operation. Overall recovery of sucrose from molasses (separator and crystallization) may go down from the high 70s to about 60% or lower (Rhoten 2004). Although the cocrystallization method appears to save production costs, it is not highly recommended. (Note: You can allow periodically some cocrystallization without much difficulty, but expect lower separator and crystallization performance.)

The **copurification of extract during the beet campaign** is not used routinely. However, it may be used because of the following two concerns:

- High color of the extract
- Odor in the granulated-refined (GR) sugar

In copurification, the extract is added to the prelimer to be limed with the diffusion juice. Then the process continues as conventional beet processing (Buckley et al. 1991). If high color and odor problems occur, the standard liquor can be treated by activated carbon. (Note: Copurification may call for installing an activated-carbon decolorization station, which requires some investment.)

RECOVERY OF THE MDC PROCESS

The **main objectives** of the MDC process are the following:

- High extract purity
- High sucrose recovery

In addition, high extract purity results in less extract molasses production during crystallization of the extract. The MDC process can recover 90+% of sucrose in molasses in the form of extract. This is called **separator recovery**. Overall typical recovery (separator and crystalliza-

tion process) is about 78%. Out of 22% uncovered sucrose, almost 10% ends up in the raffinate (CMS, for concentrated molasses solid) and betaine, and the remaining 12% ends up in the extract molasses. (Note: The 78% recovery represents an efficient MDC and crystallization operations, corresponding to the production of extract with 92% purity at the separator recovery of 90%, and production of extract molasses with 60% purity at the crystallization recovery of 87%.) For recovery calculations, see Section 2 of Chapter 1.

To calculate sugar produced per every 100 t of sugar introduced into the factory in beet, you have to multiply 15 (typical percentage of sugar loss to molasses in beet operations) by 78%, which is $15 \times 78\% = 11.7$ t. This increases the sugar recovery of the factory from the traditional rate of about 80% to about 92%, which is a significant improvement. Following is a rough comparison of performances of a factory without and with the MDC process:

Factory without MDC	% Recovery
Sugar recovered	80
Sugar to molasses	15
Sugar to pulp	2
Sugar to carbonation-lime residue	1
Sugar unaccounted	2
Total	100

Factory with MDC	% Recovery
Sugar recovered	92
Sugar to molasses	3
Sugar to pulp	2
Sugar to carbonation-lime residue	1
Sugar unaccounted	2
Total	100

BY-PRODUCTS OF THE MDC PROCESS

The **by-products** of the MDC process are raffinate and betaine. **Concentrated-liquid raffinate** (also called **CMS**, for concentrated molasses solids) has the following typical composition:

Component	% on *DS*
Salts	34.0
Protein	24.0
Sucrose	18.0
Raffinose	5.0
Nitrogen compounds	3.5
Amino acids	0.5
Others	15.0
Total	100

CMS is usually sold at about 60% dry substance (*DS*) and used as a valuable liquid feed supplement.

Betaine (trimethylglycine or glycine betaine) is a nitrogenous compound, existing in sugarbeet in higher concentrations than in any other plant. Betaine is water-soluble and chemically very resistant; therefore, it is not eliminated during processing and ends up mostly in molasses (about 5% of the solids in molasses). It is the second most valuable component of the molasses after sucrose. Molasses-desugaring systems operating in a three-fraction mode can recover considerable amounts of betaine.

Concentrated-liquid betaine has about 50% purity and is sold in the form of a liquid at about 60% *DS*. Betaine "as fed" contains approximately 30% betaine (60% × 50% = 30%).

The typical composition of liquid betaine is as follows:

Component	% on *DS*
Betaine	50.0
Amino acids	10.0
Organic anions	10.0
Salts (ash)	6.0
Sucrose	2.0
Raffinose	1.0
Invert sugar	1.0
Others	20.0

In the United States, betaine is used primarily in the animal feed industry (about 75%) as a *supplement and a feed-intake enhancer* (particularly, in chicken and turkey feed). In general, betaine is used in the following industries:

- Poultry
- Food (as a digestion enhancer)
- Citrus (as a sweetness enhancer)
- Imitation crab meat (as a flavor enhancer)
- Fermentation (as a fermentation enhancer)
- Fish (0.5 to 1.5% of feed, particularly, in salmon and trout)
- Cosmetic (as a skin moisturizing agent in lotion and as a hair conditioner)

Betaine facilitates water uptake and protects macromolecules in plant cells under stress (during freeze and thaw). The Audubon Sugar Institute (ASI) applied diluted betaine on sugarcane fields, mixing with surfactant and applying it at 5.5 kg/ha (5 lb/acre) of cane farm. ASI results indicate that betaine-treated plants grow faster than untreated ones, resulting in increased sugar yields. Betaine also improved the survival of young plants under freeze and thaw conditions (W. H. Kampen, 2000).

INSTALLATION OF THE MDC PROCESS

The **installation** of the MDC system creates better results when it proceeds in two or three phases. A phased-in system develops:

- Operating confidence
- Immediate return on investment
- Learning opportunities for personnel

- Financial sources for completion of the project
- Efficient ways to include the MDC technology into the existing factory

When incorporating the MDC process into an existing sugar factory in three phases, the installation of the juice-softening process (Chapter 6) can be the best starting point because:

- Both systems are heavily based on *ion-exchange resin technology*, but the juice-softening process is a smaller project than the MDC process and requires less investment.
- The juice-softening process is simpler to integrate into the existing factory.
- The juice-softening system is easier to operate than the MDC process.

The installation of thin-juice softening in the first phase gives sugar technologists the opportunity to learn about resin technology and time to optimize the softening operation to produce soft molasses. In addition, it gives an immediate return to the factory in terms of increased factory efficiency and reduced scaling in the evaporators. Most importantly, the installation cost for juice softening is about one-tenth the cost of a molasses-desugaring project.

The second phase might include the installation of storage tanks for extract, raffinate (CMS), and molasses. The third phase would then include the installation of the remaining equipment, such as the chromatographic columns, the evaporators for extract, raffinate and betaine, and the filters.

ECONOMY OF THE MDC PROCESS

The **price of molasses** is about one-sixth (in the United States) to one-twelfth (in some countries) that of granulated-refined (GR) sugar. The value of sugar in molasses (contains about 50% sugar) counts for one-third (in the U.S. market) to one-sixth (in some other markets) that of GR sugar. Therefore, it is more profitable for the sugar producers to desugar molasses and sell it as GR sugar than to sell it as is. However, the fact that sugar in molasses is valued less should not be the only justification factor for installing the MDC process—other important factors should be evaluated because:

- The installation cost of the MDC process is high
- The financial and operational success from individual installations ranges from excellent to unsatisfactory

Some sugar companies in the United States installed the MDC process and were so satisfied that they installed a second plant. Other companies had to shut down their MDC operation due to operational difficulties and low profitability (e.g., Fremont and Hereford factories in the United States had to shut down their desugaring plants a few years after installation). Therefore, feasibility conditions may differ and should be studied when justifying MDC installation. The basic factors in justification of the MDC installation are the following:

- **Sugar regulations:** Verify to see whether sugar is a subsidized product.
- **Difference in prices of sugar and molasses:** The prices of sugar and molasses do not fluctuate much because the price of molasses usually follows that of sugar (if the demand for molasses does not increase). In particular, *the higher the difference in prices of sugar and molasses, the more MDC installation is justified.*

- **Agreement between sugarbeet growers and processor:** This subject needs particular attention because some standard contracts call for payment to growers based on a portion of profits from sugar and by-products. In **cooperative sugar companies** (see Section 1 of Chapter 1 for more information), however, the profit always goes to the grower.

The main justifications for the MDC installation include analyses of:

- Production costs of sugar made from beet and from the molasses (the MDC process)
- Integration of the MDC process into the existing factory
- Availability of sufficient molasses
- System's competitiveness

The **production costs of sugar** from molasses, in general, must be lower than the costs from beet to justify the installation of the MDC project. Sugar prices vary considerably from country to country because of a variety of regulations and issues, so the production costs of sugar from molasses must be compared to the market price of sugar in a particular country (not in the world market). To some extent, the production costs of sugar depend on energy usage. In the MDC operation, evaporating the product (extract) and particularly the by-products (CMS and betaine) are the highest cost items. Therefore, the system that uses less elution water and produces by-product fractions with a higher concentration is more cost effective. (Note: Low water usage will usually result in lower sugar recovery and lower extract purity; therefore, water usage must be balanced with sugar recovery.)

Integration (inclusion) of the desugaring plant into an existing factory or installation of it in a new beet-sugar factory is an important topic. The sugar factory must make certain production alternations to facilitate its operation with the desugaring plant. Most installations are integrated into the factory operation utilizing factory boilers, evaporators, and technical personnel to reduce operating costs. Most beet factories that integrated the MDC process into their system have not changed their traditional beet-processing method. Applexion suggests a newer method of integration into the process when a new sugar factory is built. This method uses the MDC process for recovery of sugar from the low-green (B) syrup instead of molasses (Francis et al. 2005).

The **availability of sufficient molasses** to run the desugaring operation for a considerable amount of time every year affects the investment payback. The availability of molasses determines the size (capacity) of the MDC plant to be constructed. Molasses-desugaring plants are designed to process 300 to 600 t of molasses per day. In general, the desugaring plant should be designed for 200 to 300 days of operation per year if no restrictions exist on either environmental regulations and/or on operating days of the facility.

The minimum amount of molasses on hand must be enough to operate the MDC for more than 150 days per year. The factory's access to this minimum quantity of molasses is an important factor because some factories do not produce enough molasses. Molasses is obtained from one or a combination of the following sources:

- From the factory with the MDC process
- From the factory with the MDC process and member factories without the MDC process
- From the market at an additional purchase price

As of early 2006, five U.S. companies (total of 8) are equipped with desugaring plants and all five are multiple-factory companies, receiving molasses from their member factories. Amalgamated Sugar operates three factories with two MDC plants (Nampa and Twin Falls factories); American Crystal Sugar operates five factories with two MDC plants (East Grand Forks and Hillsboro); Michigan Sugar has four factories with one MDC plant (Monitor factory); Southern Minnesota cooperative operates three factories with two MDC plants (Renville and Mendota factories); and Western Sugar operates five factories with one MDC plant (Scottsbluff factory). None of the U.S. single-factory companies (Minn-Dak farmers cooperative, Sidney Sugars, and Wyoming Sugar) are equipped with MDC process.

In general, the factory interested in the MDC installation should have access to enough molasses. For example, a factory with 6000 t/day beet-processing capacity produces about 300 t/day molasses (at 5% on beet). If this factory has enough beets to run the factory for 150 or more days a year, the factory produces enough molasses to operate the desugaring operation for 150 days at 300 t/day. When a factory does not make enough molasses but can obtain it from a member factory or buy it from the market, that factory might still profit from having the MDC process if other requirements are met.

When determining whether to go ahead with any technical project, the **return on investment** (ROI) is the most important factor. Table 8.1 evaluates a desugaring plant with a processing capacity of 350 t/day molasses. Table 8.1 also shows the effect of the total molasses processed on the ROI. (Note: This evaluation is based on net prices of sugar, molasses, and CMS (concentrated molasses solids) and energy price in the United States as of early 2006 when the prices of sugar and molasses increased considerably and the price of oil was at a hight level of about $70/barrel.

As shown in Table 8.1, the ROI is 5.7 years for a 150-day operation. When the factory has more molasses and can operate the desugaring plant for 200 days, the ROI decreases to 4.3 years. And at 250 days of operation, the ROI gets to an impressive figure of 3.4 years. Note: The calculations are based on a two-fraction separation. The three-fraction operation results in an even better ROI because betaine recovery and its sale (betaine price in early 2006 was about $260/t in the U.S. market) add considerably to the revenue generated by the MDC process. Note that the installation costs given in Table 8.1 are for a three-fraction MDC and juice- and molasses-softening equipment.

ROI can be estimated by multiplying the amount of expected additional sugar to be recovered by the unit difference in its value as sugar and as molasses. Operating costs must also be calculated and compared with ROI to determine the best desugaring system.

A **system's competitiveness** refers to selecting the most favorable and cost-effective system from suppliers of the MDC process. The right system is one that will enhance best the economics of installing and operating the process.

A **confidential agreement** between buyer and supplier is the starting point for comparing different MDC systems offered by different suppliers. The agreement should be nonbinding as to the purchase of a system.

In a comparison of different systems, the best result can be obtained by setting appropriate **benchmarks** (standards) for different features of the MDC process. Among such benchmarks would be the following:

- Production costs
- Installation costs

- Return on investment
- Operating complexity
- Extract purity (should be 90+%)
- Sucrose recovery (should be 90+%)
- Ways of integrating into the existing factory
- Availability of operation in three-fraction mode
- Betaine recovery (in case of operation in three-fraction mode)
- Extract concentration (depends on the feed concentration and elution water used)
- Extract color (should be reasonable to produce sugar without decolorization station [extract color compared with color of feed molasses can be used for color comparison of the systems])
- Column design (sixe, thickness, and welds of the columns and internal distribution headers should be suitable to withstand the pressure and material loads encountered during operations)
- Energy costs (different systems produce extract and byproducts in different concentrations, which require different amounts of energy to be concentrated)
- Resin loading rate (tons molasses/m^3 resin per day)
- Pressure drop across the resin-bed height
- Feed distribution design in the separator
- Feed handling and dilution systems
- Elution water requirement
- Resin replacement cost
- Resin life expectancy
- Required resin cost
- Control equipment
- Analytical load
- Labor costs

Table 8.1 Evaluation of the MDC Process

Processing period (days/year)	1	150	200	250
Price/ton				
Sugar	$555.00			
Molasses	$105.00			
Molasses freight	$5.00			
Rafinate	$28.00			
Extract molasses	$80.00			
Molasses				
Processed (t)	350	52500	70000	87500
DS	80.0%			
Purity	60.0%			
Recovery				
Separator	90.0%			
Crystallization	87.0%			
Overal	78.0%			

Table 8.1 Continued

Processing period (days/year)	1	150	200	250
Extract:				
DS	70.0%			
Purity	92.0%			
Produced (t)	234.8	35217.4	46956.5	58695.7
Rafinate (CMS)				
DS	60.0%			
Purity	13.0%			
Produced (t)	214.6	32183.9	42911.9	53639.8
Extract Molasses				
DS	80.0%			
Purity	60.0%			
Produced (t)	41.1			
Sugar				
Produced (t)	131.5	19731.6	26308.8	32886.0
Production costs ($/t molasses processed)				
Molasses	$105.00			
Shipment	$5.00			
Energy	$30.00			
Utilities	$5.50			
Processing materials	$2.50			
Resin	$2.00			
Crystallization	$3.00			
Labor	$5.00			
Total cost	$158.00			
Cost				
Tons sugar produced/t molasses	0.38			
Production costs/t sugar produced	$420.39			
Production costs/cwt sugar produced	$21.02			
Revenue/day				
Revenue/t sugar produced	$134.61			
Revenue/t molasses processed	$50.59			
from sugar	$28,078			
from rafinate	$6,008			
from extract molasses	$3,287			
Net	$37,373	$5,605,910	$7,474,547	$9,343,184
Installation costs				
Molasses desugaring	$25,000,000			
Thin juice softening	$4,000,000			
Molasses softening	$3,000,000			
Total	$32,000,000			
Return on investment (year)		**5.7**	**4.3**	**3.4**

Two additional features that should be compared are the following:

- **Combining the MDC and softening technology**: Most MDC suppliers sell thin-juice softening technology as well, so the best choice is when the supplier offers the best of both technologies
- **Guarantee**: The performance guarantee by supplier should be reasonable
- **Local workforce capability**: The MDC equipment needs to be installed by experienced contractors

Thus far, the most important factors affecting the MDC process were described. Now you need a comparison method to arrive at a firm conclusion for choosing the MDC supplier that offers the best system. The **weighting method** is recommended for an unbiased comparison of the systems. This method compares the **weights** (greatness) of the factors affecting the process when some factors have a greater weight (effect) on the performance and economics of the process than do others (see also subsection of "Weighted Average" in Section 2 of Chapter 12).

For the weighting method, follow the steps below:

1. Set up a tabular list (for each supplier) consisting of three columns
2. List all the features of the MDC process in the first column (see Table 8.2)
3. Weight (rate) the features in the **weighting** column by giving 3 to the more important features and 2 or 1 to less important ones (note that some features may have relatively greater importance to one sugar producer than to another)
4. Rank (prioritize) the features in the **ranking** column by giving 1 if the system has an advantage over the other systems and 0 if it has a disadvantage (if there is no difference between the system under evaluation and other systems, each system receives 1 in the evaluation column)
5. Score the system in the **score** column by multiplying the weighting value (1 to 3) by the ranking value (0 or 1)
6. Add the scores to come up with a **total score**
7. Repeat steps 1 through 6 for each system separately
8. The system having the highest total score is the system of choice (see the upcoming Note)

Table 8.2 Example of the Weighting Method

	System 1		
Factors	Weighting	Ranking	Score
Installation costs	3	0	0
Production costs	3	1	3
Return on investment	3	1	3
Matching MDC and softening technologies	3	0	0
Resin-life expectency	2	1	2
Total score			

Note: Table is not finished

NOTE

Depending on the integration the MDC plant into the factory (existing or when a new factory is built) and other conditions, one system can work the best for one sugar producer but not be the best system of choice for another sugar producer.

CANE-MOLASSES DESUGARING

Cane molasses contains particular components that make it difficult and less economical to desugar. The eight main differences between beet and cane molasses are the following:

- No betaine in cane molasses, which negatively affects the economy of the desugaring process
- Lower sucrose content (about 40% on solids in cane molasses versus 60% in beet molasses)
- Higher nonsucrose content (60% on solids in cane molasses versus 40%)
- Higher invert sugar content (about 10% in cane molasses versus 2%)
- Higher suspended solids (about 5% in cane molasses versus 0.5%)
- Higher hardness (20 to 100 *mEq* in cane molasses versus 5 to 40)
- Much higher gum content in cane molasses
- Lower filterability rate in cane molasses
- Higher viscosity in cane molasses

Because of the differences between beet and cane molasses, the cane-molasses desugaring is impractical. However, Applexion in France, ARI and Audubon Sugar Institute in the United States, MCI in Japan, and Idreco in Italy are involved with research in the application of the **syrup desugaring by chromatographic** (SDC) **process** after the syrup is softened by resin and filtered by the membrane technology (see Section 10 in Chapter 3). The SDC process produces two fractions: extract (high in sugar) and raffinate (high in ash and invert sugar). In average, it can increase the sugar yield of the factory by 6 to 7% (Kochergin et al. 2000). Considering favorable results, the SDC process should not be too far from implementation in the cane-sugar industry.

Note: Readers interested in more information on the desugaring process on molasses and syrup can refer to Saska et al. (1993), Saska and Lancrenon (1994), and Kochergin et al. (2000).

OPERATING PROBLEMS

Most of the problems encountered in the **molasses desugaring by chromatographic** (MDC) **process** are resin related and were explained in Chapter 6. Overall, the MDC system that is properly designed has few mechanical problems. This is because the MDC systems are composed of non-moving parts with the exception of the pumps. The main issue with the MDC systems is **differential pressure** (DP) fluctuation. One of the reasons for DP fluctuation is the higher sugar content of the feed. This problem is greater in the cells just prior to extract removal

because sugar concentration is increasing from the time the feed molasses is introduced until the sugar is discharged as extract. The following are there important points that must be followed thoroughly in the MDC operations to prevent DP fluctuation:

- **Soft molasses**: Feeding the separator with soft molasses (the molasses with hardness content equal to, or less than, 3 *mEq*/100 *DS*) is the most important factor in the MDC operations.
- **Filtration**: Molasses fed to the separators must be filtered thoroughly using pressure-leaf filters with precoating and body feeding to completely remove suspended solids. Some molasses filters better than others. Generally, freshly produced molasses (particularly with a low hardness content) filter better than stored molasses.
- **Deaeration** (degassing): Filtered molasses must be degassed to remove air bobbles to prevent flow channeling.

Improper follow up of the preceding operations causes resin clogging in the separators and may ultimately shut them down for corrections. Backwashing the resin bed to remove the suspended solids is the best way to correct the DP fluctuation and the plugging problem.

To optimize the MDC performance, sometimes some adjustments on water-to-feed and extract-to-raffinate ratio are required. Correct adjustments not only improve the sugar recovery of the system but also the color of the extract and, consequently, the color of the sugar produced from the extract. To express the color efficiency, **separator color removal** (SCR, the ratio of color of feed molasses to color of extract) and **overall color removal** (OCR, the ratio of color of feed molasses to color of sugar) can be used. For example, if color of molasses to the separators is 40000, color of extract produced is 8000, and color of the sugar produced from the extract is 40 (all color values are in IU), the SCR is 5 and OCR is 100, which indicate a proper operation.

In particular, the operations of the MDC process is complex and cannot work well by simply going "by the book", it requires a great deal of technical knowledge and experience. Usually, the suppliers of the MDC technology provide adequate training courses for the technical employees of the buyer before the buyer starts operating the MDC plant.

END-OF-THE-CHAPTER NOTES

- A suitable system of the **molasses desugaring by chromatographic process** (MDC process) can offer the following:
 - Sugar recovery through separation (90+ %)
 - Sugar recovery through crystallization (78+ %)
 - Lower production costs than sugar made from beet
 - A reasonable return on investment (less than five years)
 - An extract fraction with low color and high purity (90+ %)
- A carefully selected MDC plant, after much comparative selection, and smartly integrated into an existing factory can have the following positive effects:
 - Increase income and make the beet-sugar factory more secure in the sugar marketplace
 - Help the sugar industry to stay competitive against the substitute sweetener industry
 - Produce sugar at a lower production cost than sugar produced from beet
 - Increase the sugar yield (extraction) of a factory by 10+%
 - Desugar molasses without environmental pollution
- The U.S. beet-sugar industry has improved considerably over the last two decades because of the cost effectiveness of the MDC process. (However, all the preceding advantages are achievable *only if a suitable MDC system is chosen*.)
- In the next chapter, you will become familiar with another technology (Refining Raw-Cane Sugar in a Beet-Sugar Factory) that can improve the sugarbeet processing and efficiency of a factory.

CHAPTER 9

REFINING RAW CANE SUGAR IN A BEET-SUGAR FACTORY

SECTIONS

- Properties of Raw Cane Sugar
- Purchasing Methods of Raw Sugar
- Refining Options
- Applying the Options
- Basic Refining Operations
- Economics of Raw-Sugar Refining

ABOUT THE CHAPTER **Chapter 9** discusses the refining process of **raw cane sugar** (later raw sugar) in a beet-sugar factory. Refining raw sugar puts the beet factory in a two-mode operation: beet processing and raw-sugar refining. The factory must be modified moderately to be able to refine raw sugar.

Raw sugar can be coprocessed with beet during the beet campaign or processed in a stand-alone (straight) operation during the beet intercampaign. This chapter describes both options. It also explains the modifications that must be made, equipment requirements, and the major limiting factors associated with refining raw sugar in a beet-sugar factory.

To make this chapter useful also to the cane and refining industry, the conventional refining process used by sugar refineries is briefly described. Particular attention is paid to the differences between **partial liming** (used by cane processors) and **complete liming** (used by beet processors). The economics of a refining operation in a beet-sugar factory, particularly when the factory is equipped with the molasses-desugaring process by chromatographic process (**MDC process**) is described as well.

Beet-Sugar Handbook, by Mosen Asadi
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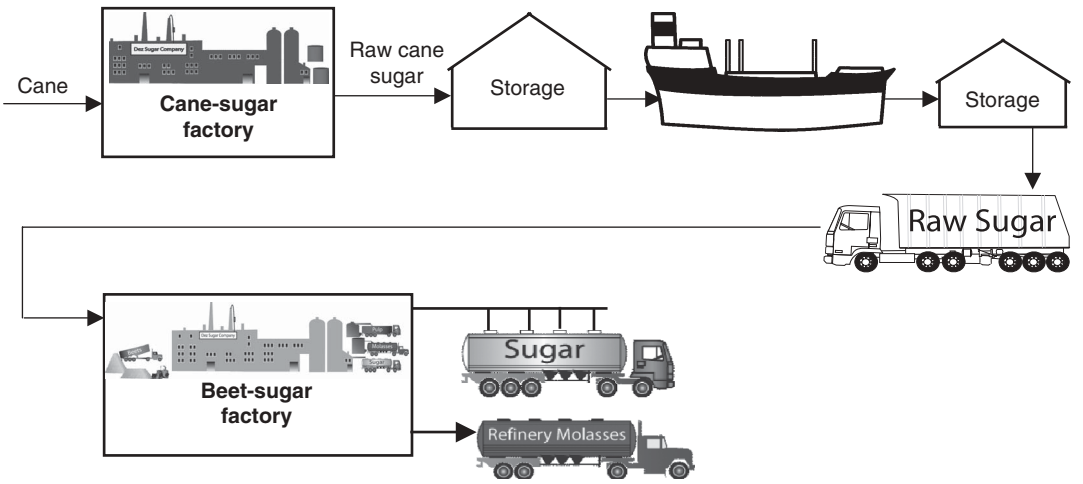


Figure 9.1 Raw cane-sugar transport to a beet-sugar factory

The refining process in the sugar industry is defined as purification of raw sugar by remelting, purifying, and crystallization. **Raw** (nonrefined) **sugar** consists of sucrose crystals covered with a thin layer of syrup. Raw cane sugar (later raw sugar) contains 1 to 4% nonsugars (purity of raw sugar is 96 to 99%). The refining process removes nonsugars and color from raw sugar. **Granulated-refined** (GR) **sugar** and **refinery molasses** are the product and by-product, respectively, of the refining process.

Refining raw cane sugar in a beet-sugar factory puts the factory in a two-mode operation:

- Beet processing
- Raw-sugar refining

Raw sugar is refined in a beet factory for a number of reasons:

- Increase profitability
- Develop a more-for-less system
- Extend the equipment-utilization period
- Make equipment perform more than one duty
- Protect the factory when it does not have enough sugarbeets
- Make the beet-sugar industry more secure in the world sugar market

The refining process is helpful to a beet factory particularly when it processes low-quality beets because raw sugar can be mixed with thin juice to increase the purity profile over the entire process. Raw-sugar refining is a *high-volume, low-profit operation*, which is not justified unless the following three conditions are met:

- The beet factory is located close to a waterway or a raw-sugar factory because transporting raw sugar to an inland factory is costly
- The price of raw sugar is reasonable
- The production volume is high

Before we get to the details of the refining process, it is important to know about the properties of raw-cane sugar and the payment methods for its purchase.

PROPERTIES OF RAW CANE SUGAR

Raw cane sugar consists of sucrose crystals covered with a thin layer of low-purity syrup. This syrup (called **syrup film** in the cane industry and **mother liquor** in the beet industry) has a much lower purity (55 to 70% depending on the raw-sugar quality) than crystals (above 99%). The amount of mother liquor in high-quality raw sugars is about 5%.

The color of raw sugar ranges from yellow to dark brown. Invert sugar, ash, organic compounds, coloring materials, and moisture are the main nonsugars (impurities) in raw sugar. The most important physical properties of raw sugar are crystal size and filterability.

The following is the composition of typical raw and refined sugar:

	Raw	Refined
Sucrose (%)	98.0	99.95
Invert (%)	0.6	0.008
Ash (%)	0.3	0.007
Organics NS (%)	0.1	0.015
Moisture (%)	1.0	0.020
Color (IU)	1000–5000	10–50

Invert Sugar

The main difference between raw cane sugar and raw beet sugar is the invert-sugar content, which is high in raw cane sugar (up to 1.5%) and very low in raw beet sugar (about 0.05%). The amount of invert sugar present in raw sugar is an important factor in the purchasing price of raw sugar and its refining workability in the factory. Depending on the purification method, invert sugar can be eliminated completely, similar to beet processing or partially, the method that cane sugar refiners use. In the case of complete elimination, invert sugar ends up in carbonation-lime residue (CLR). With partial elimination, the remaining invert sugar ends up in molasses.

Ash

Most **minerals** in raw sugar are in the form of soluble and insoluble salts of organic and inorganic compounds called **ash** content. High ash content does not have much effect on raw-sugar storage but is undesirable from a manufacturing point of view due to its **melassigenic effect** (molasses-producing actions). Raw sugar with high ash and other nonsugars produces high-viscosity juice, which causes filtration problems. Ashes, particularly Si_2O_5, not eliminated during purification create **scale** in the evaporators and vacuum-pan crystallizers.

Organic Nonsugars

Most **organic nonsugars** in raw sugar have a high molecular mass. Starch, dextran, and **colloids** (e.g., pectin, dextran, colorants, decaying beet particles, and microorganisms) are the main reasons for filtration problems, increase in filter-aid usage, and decrease in refining capacity. The types of organic nonsugars are different in raw sugar from beet and cane.

NOTE

- **The fruity taste of raw cane sugar is due to the organic esters and other aroma-type substances in the sugarcane.**

Color

The properties and amount of **coloring substances** (colorants) in raw sugar are important. **Very low color raw sugar** (VLC raw sugar) is attractive to refiners because it may let them omit the **affination** (the process of mixing raw sugar or low-purity massecuite with high-purity syrup and then centrifuging it to improve the raw-sugar quality) step. Raw sugar loses a considerable amount of its color during affination (more than 50%).

Moisture

Raw sugar with a high-moisture content cannot be stored because it is susceptible to microbial activity and sugar hardening during storage. The sugar-refining industry uses the ratio of water content (W) to nonsugars (NS) to calculate the **safety factor** (SF):

$$SF = \frac{W}{NS} \times 100 = \frac{W}{100 - (S + W)} \times 100 \tag{9.1}$$

Here, the S (sucrose content) refers to sucrose measurement by polarimetry, called **polarimetric sugar** or pol.

EXAMPLE 1

Given:

Sugar content (S) of raw sugar	98.0%
Moisture content (W) of raw sugar	0.5%

Calculate:
Safety factor (SF)

$$SF = \frac{W}{100 - (S + W)} = \frac{0.5}{100 - (98.0 + 0.5)} \times 100 = 33\%$$

NOTES

- **A good-quality raw sugar requires an SF less than 25%. There is a risk of deterioration when the SF is between 25 and 35%. Sugar having an SF above 35% is unsuitable for storage.**
- **Raw beet sugar has a higher SF (up 45%) than cane because of its low invert-sugar content.**

Crystal Size

A suitable raw sugar has uniform crystals from 0.6 to 1.0 mm, preferably 0.8 mm. The grain size is determined by screening washed sugar through a 28 mesh screen.

Recovery

Based on the quality of the raw sugar, you can calculate a **theoretical** (expected) **recovery** (yield). The recovery (R) is the percentage (on mass base) of the refined sugar produced per raw sugar processed. Sucrose (S), invert-sugar (I), and ash (A) content are used to judge the recovery of raw sugar. Different countries use different formulas for recovery estimation, as follows:

Country	Recovery (% on raw sugar)
Great Britain	$R = S - (3 \times A) - (2 \times I)$
Holland	$R = S - (5 \times A) - (2 \times I)$
France	$R = S - (5 \times A) - (1 \times I)$
Germany	$R = S - (5 \times A) - (5 \times I)$
Czech Republic	$R = S - (5 \times A)$
United States	$R = S - (2 \times I + 4 \times A)$ or $(S \times 2) - 100$

NOTES

- **Ash-content is measured by conductivity, and sucrose content by polarimetry.**
- **The sucrose loss to refinery molasses in refining operations usually ranges from 1.5 to 3.0% on sugar (see Example 2). This means that the recovery of the refining operation is *about* 97.0 to 98.5%, considering other losses (e.g., to filtration process) are minimal.**

PURCHASING METHODS OF RAW SUGAR

In almost all countries, the price of raw cane sugar is calculated based on 96% sucrose content (polarimetric sucrose, ***PS***). Raw sugar above the base level gets credit, and sugar below the limit is penalized. In the United States, the refiners pay a premium up to 98.8% and deduct for sugars that contain less than 96% sucrose. In addition to the sugar content, moisture, ash, crystal size, dextran content, and color are included in the premium (or penalty). The adjustments are different according to the country.

NOTES

- **VHP sugar (very high-pol sugar) is high in sucrose content and coated with high-purity molasses to lower the raw sugar purity. The sugar is coated because some countries have restricted tariffs that do not allow them to buy sugar with a sucrose content above 98.8.**
- **Some raw-sugar producers produce VLC sugar (very low color sugar), which gets a premium price because of its low-color content. The refining costs for VLC sugar is lower because the refiner can eliminate the affination and decolorization step from the refining process.**

REFINING OPTIONS

The beet-sugar factory has three possible options for processing raw cane sugar:

1. Coprocessing (processing sugarbeet and raw sugar together)
2. Straight refining for a short period
3. Straight refining for a long period

Each of these options has advantages and disadvantages in terms of the equipment and modifications necessary to convert raw sugar into granulated-refined (GR) sugar.

1. Coprocessing

In a coprocessing operation, the factory processes sugarbeet and raw cane sugar together during the beet campaign. The quality of the raw sugar and sugarbeet determines how much of these two raw materials can be processed. Typically, a beet factory can coprocess raw sugar up to 50% of its total GR sugar production (from beet and raw sugar) without problems. For example, if a factory produces 800 t sugar a day from beet, it can process up to 400 t raw sugar. The amount above the sugar-end capacity is sent to the **thick-juice storage tank** where it is stored until being processed during the beet intercampaign. To coprocess, the factory must modify some stations and invest in equipment and storage. The following are the minimum requirements:

- Construct a raw sugar receiving station
- Install a melter for the raw sugar
- Expand the filtration capacity
- Install juice storage tanks

The coprocessing operation (see Figure 9.2) can be outlined as follows:

1. The raw sugar is melted in the melter in heated sweet water, which is mostly supplied by second-carbonation and standard-liquor filters, to the desired dry-substance content (see the following Note).
2. The **remelt syrup** (called **melt liquor** in the refining industry) is screened to eliminate fiber, lint, and other particles that are usually present in raw cane sugar.
3. The syrup is mixed with diffusion juice from sugarbeet processing.
4. The juice is limed and gassed (refers to CO_2 gas) as in the conventional process. The amount of lime (CaO) for purification is almost the same as in conventional operation (about 80% of nonsugars entering the purification station—see Section 10 of Chapter 3 for more information on the lime requirement).
5. From this point on, almost no other changes to normal sugarbeet processing are necessary. Some changes in the crystallization scheme, such as switching from three-stage to four-stage crystallization or backboiling (see Section 17 of Chapter 3), might be made because the feed to the first stage of crystallization has a higher purity (96.5 to 99.5 depending on the raw sugar quality) than that of beet syrup (about 92%). In addition, if the amount of raw sugar processed is high, resulting in a juice with high color that purification cannot handle, the factory might add a **decolorization station**.

The advantages of coprocessing compared with options 2 and 3 are:

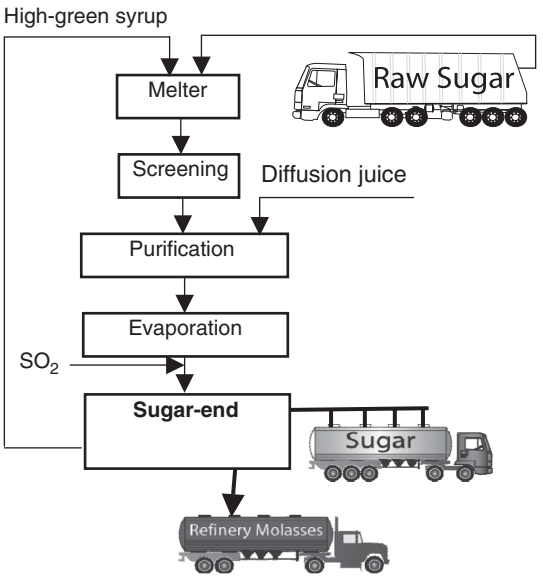


Figure 9.2 Coprocessing raw sugar with diffusion juice from beet

- High benefit
- Low investment
- Low production cost

The disadvantages of a coprocess operation are:

- **Difficult process accounting:** The beet and refining accounts should be calculated separately.
- **Capacity limitation:** Any addition of raw sugar reduces the beet-slicing rate. However, this problem can be solved by the installation of a thick-juice storage tank.

NOTES

- **In beet operation, the purification station usually handles the juice of 15 to 17% dry substance (*DS*), whereas the remelt syrup in refineries has *DS* of about 60%. In the past, beet-sugar technologists tried different concentrations of remelt. High-concentrated remelt (about 60% *DS*) is in line with cane refiners' principles but has the following disadvantages for beet processors:**
 - **Requires a purification modification to handle highly concentrated juice**
 - **Lower purification efficiency (juice carbonates worse at higher concentration)**

 On the other hand, the dilution of the remelt syrup to a lower concentration (about 30%) does not require a purification modification (an advantage) but rather more energy to evaporate the extra water (a disadvantage).
- **After melting, a screening operation is needed to eliminate cane fiber, lint, soil, suspended solids, and colloids (large-molecule polysaccharides). Poor screening creates pumping and purification problems. A vibrating screen is the most suitable equipment for this job. Note that the diffusion-juice screen in beet-sugar factories is not sufficient for the screening of remelt sugar.**

2. Straight Refining for a Short Period

The beet-sugar factory can process raw sugar alone during the intercampaign by using the last batch of lime from the limekiln for purification. Refining preferably starts right after the beet campaign and continues until the lime storage is emptied. The length of the refining period depends on the plant's daily refining and limekiln capacity.

At the end of the beet campaign, the limekiln can be left on for one or two more days to make one or two extra batches of lime. For example, a limekiln with a holding capacity of 600 tons of limestone (the size of limekiln needed for a factory with 8000 t/day capacity) can make $600 \times 56/100 = 336$ t of quick lime (based on a molecular mass of $CaCO_3 = 100$ and $CaO = 56$). This amount of lime can stay in the limekiln and be taken daily to the slaker to produce milk of lime (MOL). Suppose that the factory intends to refine 400 t of raw sugar a day. Assume that the amount of lime for purification of the remelt syrup is equal to about 4% CaO on raw sugar (in complete liming). Therefore, 336 t of lime is enough to process $336 \times 100/4 = 8400$ t of raw sugar equal to $8400/400 = 21$ days of refining operations.

In this operation, the **flue gas** (burned gas before entering the stack of the boiler) from the boilers is used as a source of CO_2 during the refining period. The amount of carbon dioxide in the flue gas is lower (about 14%) than in the carbonation gas (about 33%). This does not create difficulties because the carbonation equipment is usually designed to operate with gas containing as little as 12% CO_2. The advantages of the straight (stand-alone) method for a short period, compared with other options, are as follows:

- Lower cost for the limekiln operation than option three (straight refining for a long period) because the limekiln is not in operation during the entire refining time
- Fewer complications in process accounting because there are two separate campaigns (beet and raw-sugar processing)
- Short storage time for raw sugar compared to option 1
- Enough intercampaign time for maintenance
- Lower capital cost than option three

The disadvantages of this method are as follows:

- Uses flue gas from boilers, which has a lower CO_2 content than gas from the limekiln
- Requires an extra investment in the flue gas scrubbers

Figure 9.3 shows the straight refining with and without affination and decolorization.

3. Straight Refining for a Long Period

With option 3, **straight refining for a long period**, the factory stays longer in refining mode by letting the limekiln operate at a low speed during the entire refining period. Unlike straight refining for a short period, in this option both lime and CO_2 gas from the limekiln can be used for purification. This makes the beet factory operate similarly to the sugar refinery, with the advantage of producing its own lime. During this period, the adjustment of the limekiln to a slower rate (based on purification demand) is important because the refining needs much less lime than beet operations. However, excess lime can be used for wastewater treatment.

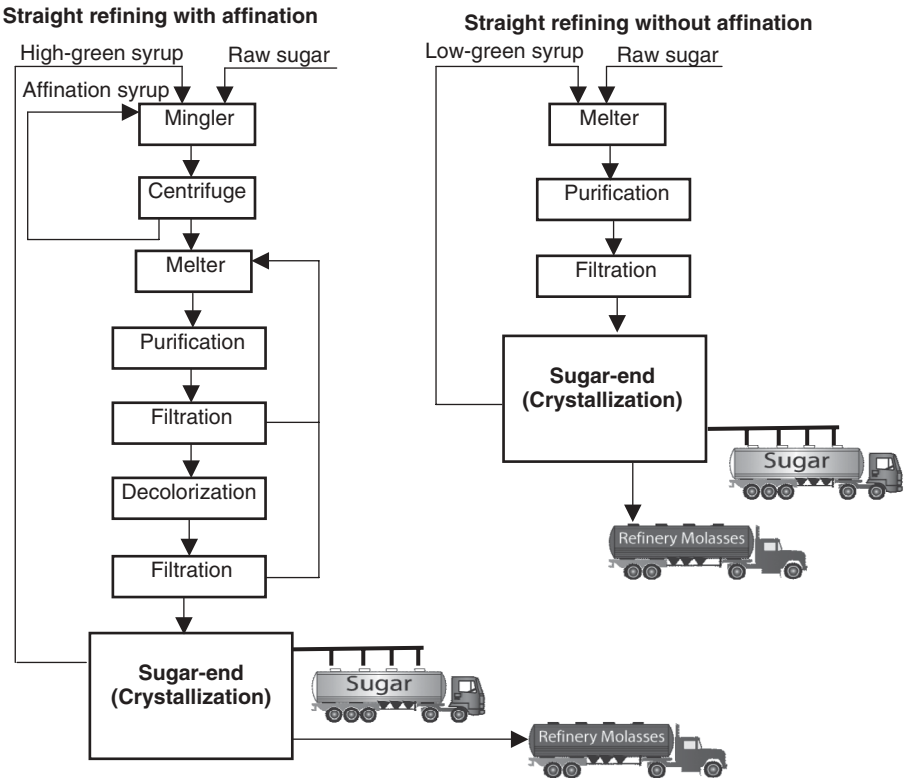


Figure 9.3 Straight refining with and without affination and decolorization

In straight refining for a short or long period, the entire operation of refining is similar to the conventional beet operation with a few exceptions. However, the capital requirement to build full-size storage for the raw sugar and the expansion expenses for the dryer and the silo for sugar storage are high.

The disadvantages of straight refining for a long period are as follows:

- Increased wear-and-tear on the limekiln
- High capital investment and long payback
- Strong dependence on the price of raw sugar
- High-efficiency cooling water needed for summer operations
- Additional capital investment required to expand the refined sugar storage
- Less intercampaign time available to maintain the factory and clean the evaporators

APPLYING THE OPTIONS

Some beet factories around the world refine raw sugar. The following are examples of raw-sugar refining in some U.S. beet factories based on the options previously described.

Option 2 (**straight refining for a short period**) was used in the Easton Beet-Sugar factory and refinery in Maine (in the United States) from 1966 to 1970 (Schwieren 2002). Easton was a factory with 4000 t/day beet and 500 t/day refining capacity. It was shut down in 1970 because of beet-growing difficulties and new farming regulations. Every year, one boatload (30000 to 35000 t) of raw sugar was refined after the beet campaign was finished. The main operations follow:

- Raw sugar was unloaded from the boat to a railcar and transferred to the factory, which was about 120 km (75 miles) from the port. The raw sugar was stored in a 30 by 100 m (90 by 300 ft) covered warehouse.
- The refining process consisted of affination, remelting to about 60% *DS*, purification, decolorization, and crystallization.
- The purification system was the same as the one used during the beet campaign, except flue gas was used for the CO_2 supply.
- To prevent higher color juice, evaporators were not used to increase the concentration of the juice. However, one effect of the evaporation station was production of steam for use in the crystallization operation.
- Syrup was decolorized by powdered-activated carbon (PAC), which was discarded after use.
- A four-stage crystallization scheme was used during the beet and refining period.

The Fremont sugar factory (a 3500 t/day beet factory) in Ohio (United States) started to modify its factory to refine raw cane sugar in 1978 (Toth 1984). A year after that, the factory was in operation for 138 days, refining raw sugar during the beet intercampaign. This proved that a beet factory can operate successfully for a longer period in refining mode with a minimum of additional equipment. The refining was shut down for 40 days during which the maintenance of the equipment was performed. The main modifications to the operations in Fremont factory follow:

- A raw sugar receiving and affination station was constructed in the existing pulp warehouse.
- In addition to the powder-activated carbon (PAC) station, a mixed-bed ion-exchange resin for juice decolorization was installed.
- The purification station was modified to handle syrup of 60% *DS*, and flue gas was used as the source of CO_2. The purification was operated with a partial invert-sugar precipitation similar to that in refineries.
- The sugar-end of the factory was adapted to a *five-stage crystallization* scheme because it was necessary to have a drop in purity from about 99% standard liquor to produce molasses with satisfactory purity. The first four massecuites, with purities of 99.8, 98.0, 96.0, and 92.0%, were centrifuged to make white sugar.

The Fremont plant refined about 500 t/day of raw sugar. The factory had two limekilns, one of which ran at low speed during refining time because about 4 t of quick lime per day was enough for purification in addition to 4 t for wastewater treatment.

In 1988, Manteca and Woodland factories (now operated by Southern Minnesota Cooperative) began to coprocess raw sugar during the beet campaign (Rhoten 2004). Satisfactory

results from coprocessing convinced management to expand the operation with straight refining of raw sugar for more than one month a year. The straight refining operation proceeded as follows:

- Raw sugar was trucked to the factory from a nearby port.
- The raw sugar was melted to a concentration of 20% without affination and treated with lime at levels more in line with beet processing than raw-sugar refining.
- The limekiln ran at its minimum rate during refining time.
- A granular-activated carbon (GAC) system was in service to reduce syrup color.
- A four-stage crystallization scheme was used instead of three.

Although the process was successful, there was not enough margin between the cost of the raw sugar and the selling price of sugar for the factory to remain in the raw-sugar refining business. Total refining costs were similar to those of a cane refinery. Sugar recovery was somewhat lower than typical refining due to a higher purity of refinery molasses.

BASIC REFINING OPERATIONS

The refining process consists of the following operating steps:

1. Receiving and storing
2. Affination
3. Melting
4. Purification
5. Decolorization
6. Crystallization
7. Drying and storage of granulated-refined (GR) sugar

Note: Some differences exist in the refining procedures in a conventional refinery as opposed to those of a beet factory. Therefore, the explanations in this section should be understood only as general guidelines.

Receiving and Storing

Raw cane sugar is produced mainly in South America, Africa, and Australia and is transported by ships with a capacity from 10000 to 36000 t. In general, the larger the ship, the lower is the ocean-freight cost. The sugar is unloaded from the cargo vessel at the dock by crane and grab bucket. It is a good idea to install a conveyor system capable of unloading a vessel in about 24 hours because the ship companies charge overtime for unloading after that period.

Almost all refineries are built next to a port and have their own dock and sugar storing facilities. On the other hand, beet factories usually do not have this advantage because most are located inland. Even if a beet factory has access to a waterway, it likely will not have a dock or a raw-sugar warehouse.

The raw sugar is transported to the factory daily by truck or railcar. Therefore, the factory must have a storage facility that can hold sugar equal to a day and half of its refining capacity (see the following Note).

Raw sugar with suitable moisture content can be stored satisfactorily in any warehouse that can keep humid air out of the factory. Some warehouses are equipped with an underfloor conveyor for conveying sugar; others use portable equipment. Raw sugar is not free flowing, so it cannot be transferred like granulated-refined sugar. Usually, front-end loaders with rubber tires are used to feed the underfloor conveyor. In addition, some warehouses are equipped with an overhead conveyor along the peak of the building. To avoid deterioration, raw sugar should be processed on a first-in, first-out basis.

NOTE

An average density of 800 to 900 kg/m^3 is used for raw-sugar storage calculations.

Affination

Affination is the process of mixing raw sugar or low-purity massecuite with high-purity syrup (mostly low-green syrup) and then centrifuging it to improve the raw-sugar quality. As stated, raw sugar crystals are covered by a thin layer of low-purity, high-color syrup, called mother liquor. The main functions of the affination process on raw sugar are as follows:

- Increases the purity of the mother liquor
- Increases the purity of raw sugar
- Decreases the color of raw sugar

With affination, the quality of raw sugar is improved at the expense of nonsucrose recycled to the raw side, causing an increase in molasses purity (the disadvantage of affination). Therefore, when the color of raw sugar is low (below 1200 IU), affination is usually omitted. Affination is performed in two stages:

- Mixing
- Centrifuging

In the **mixing step**, raw sugar is mixed (mingled) with a saturated syrup of about 85% purity and 75% *DS* in a mixer for about 15 minutes to produce a massecuite (magma). The main purpose of mingling is to loosen the syrup coating around the crystals by rubbing the crystals together to make a homogenized massecuite. Usually, the syrup used in affination is runoff from affination centrifugals (called **affination syrup** in the refining industry) or high-green syrup from the sugar-end. Roughly, the required syrup (about 75% *DS*) for affination is equal to 25% of the total mass of the massecuite. It means that for each 100 kg of the magma, 75 kg of raw sugar and 25 kg of syrup are needed.

The affination syrup should be heated to achieve optimum temperature of the massecuite (around 50°C). The temperature, *DS*, and purity of the affination syrup are important factors because the mother liquor of the magma should be kept in a saturated state. Otherwise, the crystals start to dissolve, increasing the purity of the mother liquor, which is not desired.

In the **centrifuging step**, the massecuite is directed to the batch centrifuge or continuous centrifuge to be separated into affined sugar and affination syrup. If good-quality raw sugar is used, cold water can be used for washing instead of hot water.

NOTE

The affination station is important in the refining process, particularly when low-quality, high-color raw sugar is processed. However, the beet factory can eliminate the affination process because of the availability of extra lime. Unlike cane-sugar factories or refineries, all beet factories have their own limekiln. Cane factories and refineries buy hydrated lime and use CO_2 in flue gas from boilers for carbonation. The cost of buying hydrated lime is more than the cost of producing their own milk of lime (MOL) by using the limekiln and slaker. This makes the beet factory capable of using extra MOL during purification. Using MOL in excess amounts and high-quality CO_2 during purification improves the decolorization effect and nonsucrose elimination. Therefore, the affination stage can be omitted because the main function of affination is the reduction of color and nonsugars, which is accomplished during purification.

Melting

Raw sugar (if the factory does not use the affination step) or affined sugar (the affination step is used) is melted in the melter in heated, high-purity sweet water to about 60% *DS* to produce remelt syrup (**raw liquor**, in refiner's language). The sweetwater from the filtration station is used for dilution.

Refineries use a batch melter or a continuous melter. Usually, the batch-type melter is a shallow and round, vertical tank with a perforated screen about one-third the height of the melter situated below the upper rim. The batch melter also has a few paddles attached to the shaft, which turns slowly. Steam coils in the melter regulate the temperature to about 85°C.

Purification

The literature on the purification of coprocessing raw sugar with beet juice or straight refining of raw sugar in a beet-sugar factory is poor, although this process is not new to the beet-sugar industry. Despite its high purity (above 95%), the remelt syrup still has some impurities and color, all of which have to be reduced, and it must be kept at an optimum pH to remain stable during the next processing steps (evaporation and crystallization). For these reasons, the remelt syrup is purified in the purification station. During purification, insoluble solids, gums, polysaccharides, and dissolved colorants are precipitated and removed by filtration. Some refiners use a combined purification consisting of phosphoric acid and lime by which 30 to 35% of the syrup color is removed.

Unlike cane processors, who use **partial liming**, beet processors use **complete liming** for purification of the remelt syrup. One of the important reactions taking place during liming is the destruction of invert sugar. In a complete liming process, the invert sugar is destroyed as much as possible by adding an excessive amount of lime to obtain a high pH syrup (about 12). But cane processors destroy invert sugar partially. This is the reason why raw cane sugar contains a high amount of invert sugar, which would create excessive coloring materials if allowed to decompose.

Refineries also use partial liming in refining raw sugar by using about 1% CaO on solids in the juice (equivalent to about 1.5% on raw sugar), whereas beet-sugar factories use lime at about 4% or even up to 10% on solids (see the Note below).

The main benefits of complete liming are as follows:

- Decomposes more invert sugar
- Produces juice with lower color
- Produces juice with fewer nonsugars
- Produces juice with more thermostability
- Produces juice with a higher buffer capacity
- Produces juice with less filtration and settling difficulties
- Produces juice with a lower pH drop during evaporation and crystallization
- Produces juice with a higher pH (about 9 compared with 7.3 in partial liming)
- Produces juice with lower color formation during the next steps in the process

The major benefits of partial liming are as follows:

- Saves on lime use
- Produces juice with less hardness (limesalts)
- Saves on energy because refiners use highly concentrated remelt syrup
- Does not need a limekiln, which creates a considerable savings on its installation and operations of the limekiln (cane processors and refiners buy hydrated lime from outside suppliers)
- Creates less sugar loss to molasses (invert sugar decreases sucrose solubility)
- Produces molasses with a high invert-sugar content, which can be used in producing brown sugar. This is economical because invert in brown sugar is valued at a much higher price than molasses. (Beet-sugar producers buy food-grade cane molasses for making brown sugar because cane molasses has a fruity taste.)
- Saves on capital investment (lime and carbon dioxide addition are added in one tank)

The cane and refining industries prefer partial liming, but the beet industry favors complete liming.

NOTE

The refining industry expresses lime (CaO) usage based on % solids in the juice, but the beet industry uses %CaO on nonsugar content in the juice.

Decolorization

A beet-sugar factory that employs complete liming and an appropriate crystallization scheme can produce high-quality sugar from the remelt syrup without a decolorization station. Decolorization is also not needed when polymers such as Talofloc and Talocarb are used during purification because they can decolorize the syrup by about 75%. Refer to Section 14 of Chapter 3 for more information about decolorization.

Crystallization

Remelt liquor (liquor produced from raw sugars from different stages of crystallization melted in remelt syrup) is carried to the sugar-end for further evaporation of water to produce supersaturated solution. The supersaturated liquor is seeded, and the crystals are given enough time to grow. Crystallization of remelt liquor is fundamentally the same as that of standard liquor

from beet processing (see also Section 16 of Chapter 3). But the usual three-stage crystallization scheme cannot produce reasonable low-purity molasses because of the high purity of the remelt liquor (about 99% in compared to about 92% in standard liquor).

In general, the advantages of crystallization of high-purity syrup are as follows:

- Lower retention time
- Faster crystallization rate
- Lower concentration fluctuations

The disadvantage of crystallization of high-purity syrup is that it needs more crystallization stages.

Note that **exhaustion** of the refinery molasses is not as important as in the case of beet molasses for the following good reasons:

- Refinery molasses is edible and can be used in the production of brown sugar
- The amount of refinery molasses is very small, making sugar loss too low (see the Example below)

EXAMPLE 2

Given:

Nonsugar content of raw cane sugar processed (NS_{RS})	1.0%
Dry substance (DS_M) of refinery molasses produced	77.0%
Purity (P_M) of refinery molasses produced	73.0%

Calculate:

Amount of sugar loss in refinery molasses

Amount of refinery molasses produced

In sugar technology, the *sucrose-to-nonsucrose* ratio (S/NS) is called the **melassigenic coefficient** (K_M). The K_M can be calculated from molasses purity:

$$K_M = \frac{S_M}{NS_M} = \frac{P_M}{100 - P_M}$$

$$K_M = \frac{73.0}{100 - 73.0} = 2.7$$

This means that each kg (or pound) of nonsucrose in raw sugar will carry 2.7 kg (or lb) of sugar to molasses. Then, the sugar loss to refinery molasses will be

$$2.7 \times 1 = 2.7\%$$

This is low compared with sugar losses to molasses produced from beet processing (about 15%). This means that the refinery makes *about* 100 kg of granulated-refined (GR) sugar from processing 102.7 kg of this raw sugar (considering other losses to be minimal). Note that refinery molasses has much higher purity than cane molasses (about 40%). Cane molasses (produced from processing cane) contains a large amount of invert sugar (about 10%) and other nonsugars.

We can calculate the amount of refinery molasses produced (m_M) based on the non-sucrose content of raw sugar (NS_{RS}) and molasses dry substance (DS_M):

$$m_M = \frac{100(NS \times K_M) + NS_{RS}}{DS_M}$$

$$m_M = \frac{100(1.0 \times 2.7) + 1.0}{77.0} = 3.5\% \text{ On raw sugar}$$

Drying and Storing Refined Sugar

Refined sugar is dried and stored in the same way as in a beet-sugar factory, as detailed in Section 19 of Chapter 3.

ECONOMICS OF RAW-SUGAR REFINING

Economically, the decision to refine raw-cane sugar in a beet-sugar factory mainly depends on the following:

- **Raw-sugar price**: The price of raw sugar should be reasonable to justify the refining process.
- **Transportation cost**: The beet factory that wants to refine raw sugar must have access to a waterway or to be closed to a raw-sugar factory because the transportation cost is high.
- **Processing capacity**: Refining raw sugar is a relatively low-profit operation. Sugar refineries that process nothing but raw sugar process large quantity of raw sugar because raw-cane sugar refining is not justified unless the production is large. Therefore, it is necessary to process large amount of raw sugar and to maintain a high level of operating efficiency to end up with some profit.

The profit of the refining operation improves when the factory is equipped with the **molasses desugaring by chromatographic process** (MDC process) because the refining can be considered a bonus to its routine operations. If raw sugar is coprocessed with the sugarbeet and the juice is subjected to complete liming, the molasses produced, after being soften in the molasses-softening station (Chapter 7), can be desugared in the MDC plant. In this case, the MDC process can be utilized as a recovery house for coprocessing operations when molasses purity is higher than normal. This alternative allows three-stage crystallization to be in operation during the coprocessing period. Otherwise (without the MDC process), a change from a three-stage to a four-stage crystallization may be required to produce low-purity molasses.

Note: Maintaining the mechanical integrity of a factory with a refining operation requires more capital because the factory is in operation more days. In other words, maintenance costs of at least the sugar-end will increase with increased operating days.

END-OF-THE-CHAPTER NOTE

Chapter 9 discussed how refining raw-cane sugar in a beet factory can generate extra income for the factory. Refining in a beet factory is justified if the price of raw sugar is reasonable, if the beet factory has access to a waterway or is closed to a raw-sugar factory (the transportation cost of raw sugar is high), and if the beet factory refines a large amount of raw sugar (refining is a high-volume, low-profit operation).

CHAPTER 10

ENVIRONMENTAL CONCERNS OF A BEET-SUGAR FACTORY

Written by J. L. Carlson

Adapted by M. Asadi

SECTIONS

- Pollution and Pollutants
- Pollution Control Strategies
- Wastes of a Beet-Sugar Factory
- Air Pollutants
- Liquid Wastes
- Wastewater Treatment
- Solid Wastes
- Environmental Management System

ABOUT THE CHAPTER **We** are dependent on water and air in every minute of our life. Thus, it is the responsibility of every one of us to not degrade the natural environment with solid, liquid, and air pollutants. **Chapter 10** has accordingly been prepared to cover the strategies to control solid, liquid, and air pollutants and the wastes of a beet-sugar factory. The systems for treatment of wastewater and solid wastes for reuse and release are also described (Figure 10.1 and 10.2).

The **beet-sugar factory** produces more waste products than sugar, pulp, and molasses combined. If beet-sugar producers are not diligent in caring and disposing of waste products, the wastes can pollute the natural environment significantly. It makes good business sense for producers to reduce pollution and comply with all legally required actions and limitations with

Beet-Sugar Handbook, by Mosen Asadi
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Figure 10.1 Wastewater system (Courtesy, Michigan Sugar Company)

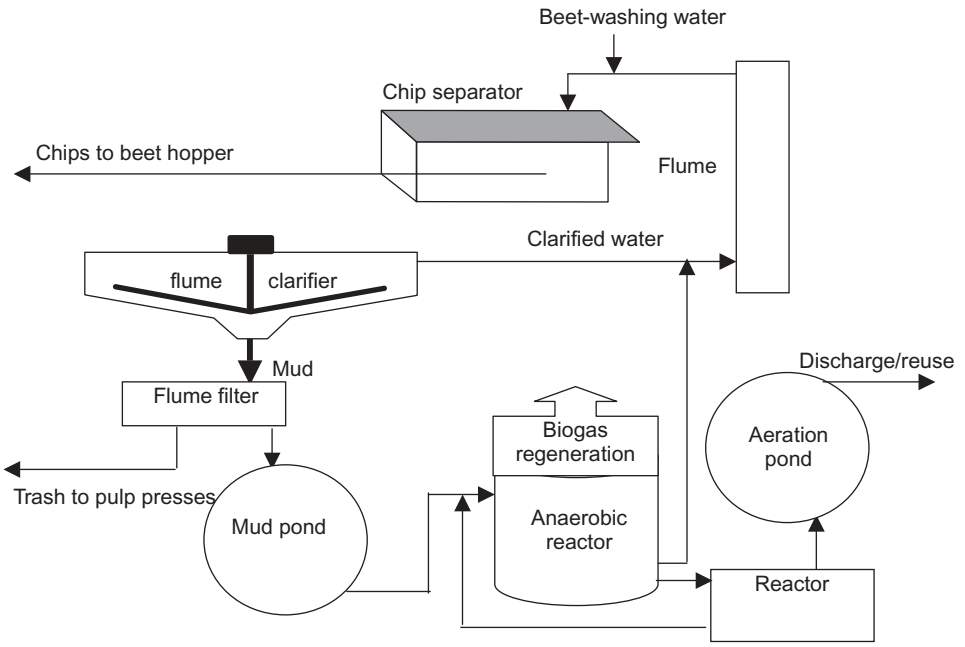


Figure 10.2 Flow diagram of wastewater treatment system in a beet-sugar factory

regard to the creation, storage, treatment, and disposal of pollutants, wastes, and hazardous compounds. Not doing so invites costly enforcement actions by the governments and lawsuits by citizens. If comprehensive pollution reduction is approached correctly, sugar producers not only be environmentally safe but also can generate considerable cost savings.

POLLUTION AND POLLUTANTS

Pollution is the action of polluting (contaminating) the environment. People pollute the environment when their activities cause a harmful change to the natural system. Some changes, however, are part of natural cycles in the **biosphere** (the part of the planet's outer shell, including land, water, and air), which means they exist apart from human intervention. For example, the granulated-refined (GR) sugar sold to the public is part of the carbon cycle. It was created from carbon dioxide and water through the **photosynthetic process** (a complex reaction in plants to produce monosaccharides by absorbing energy from sunlight) and converted back to carbon dioxide and water through cellular respiration. The beet-sugar manufacturing process does not materially affect this cycle. On the other hand, the generation of carbon dioxide, water vapor, other gases, and ash from fossil fuel combustion are not part of a natural cycle.

Beet-sugar factories are open systems, requiring exchanges with the environment. The raw materials and energy used to operate a sugar factory come from the natural environment. The products and wastes created by operating the plant ultimately go back to the environment. How beet-sugar manufacturing facilities release wastes back to the environment determines whether the process is polluting.

Generally, **pollutants** are divided into the following three groups:

- Air pollutants
- Liquid pollutants
- Solid pollutants

This type of division is somewhat misleading because pollution control for each medium is not independent of the other media. For example, by selecting a wet scrubber to remove sulfur dioxide (SO_2) from boiler stack emissions, a facility manager has chosen to transfer the SO_2 to the water and may then have to address a problem with SO_2 in the water. However, dividing the pollutants into air, liquid, and solid groups is helpful for orientation purposes.

Air pollutants fall into three categories:

- Gases
- Particulate matter (including opacity)
- Odor

Most of the **polluting gases** emitted from beet-sugar facilities are the result of steam generation, internal combustion engine operation, beet-pulp drying, lime production, noncondensable gas venting from evaporators, and wastewater treatment.

Most of the **particulate matters** (PM, tiny solid particles suspended in air) in beet-sugar facilities are created by steam generation, coal handling, sugar handling, and beet-pulp drying and handling. Most of the offensive **odors** (chemicals in the air sensed by the sensory system located in the nose) are generated by spoiled beets, beet-pulp drying, and wastewater treatment. Odor-related pollutants are difficult to deal with because the chemistry of odor formation is

too complex to indicate with simple chemical equations. In particular, **volatile organic compounds** (VOC, organic compounds that evaporate easily) and H_2S emissions from ponds are the main causes of problematic odors. Minimizing or eliminating odor sources will be discussed later.

Beet-sugar producers always have to discharge water into the environment because sugarbeets are 75% water. Most of the water evaporated when producing sugar is condensed to recover the energy in the steam, so the water from the beets is discharged as a liquid.

A variety of physical, chemical, and biological parameters are used to measure water quality. The required quality of discharged water depends on how it will affect the environment, which in turn depends on the method of the discharge, the entity that receives the discharge, and the location of the facility (most beet factories are located on or near a river).

Solids in beet-manufacturing waters come from washing and fluming beets, lime waste, wastewater treatment, air pollution control, and storm water runoff. Chemical pollutants are added by these sources as well as from condensate and cooling waters. Wastewater treatment and storm water runoff are sources of biological contaminants. The **regenerant** effluent of some resin-based processes employed by some beet factories, for example the conventional juice-softening process (discussed in Chapter 6), can also add to the volume of wastewater system.

Beet-sugar manufacturing has a variety of **solid waste** products, including dirt, wastewater sledges, rocks, weeds, beet pieces, discarded beets, discarded beet pulp, limekiln waste, precipitated calcium carbonate (carbonation-lime residue), used oil, laboratory wastes, discarded process chemicals, scrap metal, paper scrap, and coal ash. Most of the solids are not harmful, but harmful compounds can leach out of them and spread through the environment.

POLLUTION CONTROL STRATEGIES

Managers at each facility have to determine what combination of strategies makes sense for their situation. Weather, soil types, ground and surface water, facility age, current equipment, local population, local regulatory interests, costs and return on investment are some of the factors that managers must balance when selecting and implementing pollution control strategies. Pollution control strategies fall into one of the following four categories:

- Prevention
- Treatment
- Measured release
- Storage

After management has selected and implemented a strategy, plant personnel must operate and maintain the systems to economically perform the required functions.

One way to control pollution is to not create polluting compounds or conditions through energy efficiency, better controls on operations, changes in processes, and reduced wasting. Increasing energy efficiency reduces pollutants associated with energy generation. Facilities can reduce the amount of wastewater through changes in beet transport and washing.

Treatment of Wastes for Reuse and Release

Treating waste-bearing streams to reduce the pollutants to an acceptable level is often the most practical way to meet requirements. Wastewater and carbonation-lime residue from carbonation

(when it is not sold and stored in the lime pond of the factory) are two waste streams commonly treated and reused in the factory.

Wastewater treatment includes removing sugar and nutrients leached from washing the beets. Reburning carbonation-lime residue not only recalcines the material but also burns away small beet pulp pieces. Treatment for the release of waste gas streams includes removing particles, SO_2, NO_X (NO, and NO_2, collectively called NO_X), and volatile organic compounds (VOC).

Release into the Environment

Governments put strict requirements on what pollutants industries can release and how they can release them. Beet sugar producers release water through discharges into waters (rivers or lakes) or onto land. Gases are released directly through smokestacks, vents, and tailpipes and indirectly through ponds and solid waste piles. Solid wastes are applied to the land or used as fill materials. Facility managers are responsible for ensuring that the releases meet all requirements.

Storage of Wastes

Beet-sugar producers use both temporary and permanent waste storage. Most waste storage requires a governmental permit, special storage facilities, monitoring, and reporting.

Waste oil, wastewater, industrial garbage, fluorescent bulbs, batteries, dirt, and boiler ash are all examples of what beet-sugar producers store temporarily. Many facilities have their own landfills for carbonation-lime residue and boiler ash. Most facilities send at least some wastes to industrial, municipal, and hazardous waste landfills.

WASTES OF A BEET-SUGAR FACTORY

To understand the magnitude of the potential environmental impact from the manufacture of beet sugar, it is helpful to look at the type and amount of wastes and pollutants coming from each station of beet-sugar factories.

Most wastes are produced in the following steps of operations:

- Sugarbeet storage
- Sugarbeet cleaning
- Sugarbeet slicing and diffusion
- Pulp drying
- Juice purification
- Heaters, evaporators, and pans
- Sugar and by-products storage
- Wastes from steam and power production
- Process chemicals and fuels
- Other wastes

Wastes from Sugarbeet Storage

Stones, dirt, small pieces of beets, and weeds separated from sugarbeets during storage and process are known as **tare** (impurities). The tare must be handled appropriately. Usually, tare

is loaded into the beet-delivery trucks and hauled back to the fields. Under good harvesting conditions, tare is only a small percentage of the beets harvested. If growers harvest beets in wet weather, however, the amount of dirt can increase significantly.

If sugarbeet is stockpiled for temporary or permanent storage, care must be taken because it can give off offensive **odors** as the materials decompose. Storm water can carry away nutrients and contaminate ground and surface waters. **Spoiled sugarbeets** have the same issues of odor and water-soluble nutrients as the dirt in sugarbeets. If a large amount of sugarbeets is spoiled, the beet juice can kill fish if it reaches rivers or lakes.

Transporting equipment have tailpipe (exhaust-pipe) emissions, create dust from truck traffic (fugitive emissions), and have oil and fuel leaks. Companies need to maintain vehicle engines and pollution control equipment to meet the Federal Department of Transportation (DOT) and Environmental Protection Agency (EPA) requirements, as well as those from state and local regulators. Beet-sugar producers usually control dust by keeping the roads wet, or by applying calcium chloride ($CaCl_2$) or some other **dust suppressant**. Workers need to promptly report and clean up any petroleum leaks. Good storm-water-runoff control plans include directions for workers for these leaks.

Waste from Sugarbeet Cleaning

Cleaning sugarbeets has the same issues with dirt, stones, and other plant materials and beet chips that occur when piling sugarbeets. Used wash water contains not only dirt but also dissolved sugar and nutrients leached from the sugarbeets.

The **tare** (clay, sand, stone, and trash mixed with the beets) varies from below 2% to more than 8%. Chips from washing beets are highest when handling fresh, crisp beets. In northern climates, where sugarbeet freezing occurs during storage, beet skins can add significantly to the **vegetative matter** coming from the beet washer and beet flume. In addition, thawed beet tissue loses its ability to hold onto cellular juices, greatly increasing the sugar loss to the wash water.

Wastes from Sugarbeet Slicing and Diffusion

Few direct wastes are associated with beet slicing and diffusion. Good beet-washing operations minimize knife replacement. A preventive maintenance program minimizes equipment wear and replacement. Both minimize the pollution associated with the manufacture of replacement equipment and the disposal of worn or broken equipment. In a tower-diffuser operation, keeping the proper packing in the cossette mixer enhances its heat-exchanging function, thereby reducing energy consumption and its associated environmental impacts.

Microbial infection in the diffusion system has a complex environmental effect. Microbes convert sugars to acids, which reduces the pH that aids in pulp pressing. However, the acids increase the soluble nonsugar content of the diffusion juice, which increases the amount of lime, soda ash and other chemicals used in juice purification and softening. Increased **hardness** (limesalts) in the juice creates scale in the heat exchangers and evaporators' surfaces, requiring more chemical cleaning. Most of the soluble nonsugars created by microbes are not removed in conventional juice purification. They carry through to the sugar-end and increase the molasses losses. All detrimental effects on sugar processing also increase the generation of pollutants and associated costs.

If the infection is kept low, benefits can outweigh detriments, both environmentally and economically. However, managers should decide to run a low-level infection in a tower

if adequate monitoring and training are in place so that infection can be kept under control.

Wastes from Pulp Drying

Pulp handling and drying can have a number of detrimental effects on the environment and the bottom line. Decisions on the final disposition of discarded wet or dry pulp should take into consideration the nutrient content potential for storm water runoff contamination and odors. Pulp dryer stack emissions include those associated with burning fuel as well as particulates, VOCs (volatile organic compounds), CO, and ammonia from the drying process. Coal-fired pulp dryers generate ash as well.

The steam exhaust from **steam-powered pulp dryers** is condensed to capture latent heat, reducing the total air pollution from the factory. However, condensing the steam captures VOCs and particulate matter, which are added to the wastewater stream. This water is acidic and generally has enough **BOD** (biochemical oxygen demand) to require treatment before discharge. Beet-sugar factories need acids, so careful planning can turn this waste stream into a resource.

Wastes from Juice Purification

Solid wastes associated with juice purification include precipitate calcium carbonate (PCC), fines from screened limestone and coke, discarded calcium oxide, partially burned limestone, lime slaker waste, and used filter aid. Beneficial uses can be found for most of these wastes. PCC is the highest volume of these wastes, accounting for 5 to 8% on beet (% of the mass of sugarbeets sliced by the facility).

Air emissions from limekilns include hydrochloric acid (HCl), CO, CO_2, NO_X, SO_2, and particulate matter. The alkaline beet juices in juice purification remove most of the air pollutants. For example, tests of the carbonation vents at Minn-Dak Farmers factory show that juice purification effectively scrubs out pollutants. Exhaust gases venting directly from the limekiln can contain significant amounts of pollutants.

In lime–carbon dioxide juice purification, ammonia (NH_3) is released from some compounds found in the **in-process products** (juices, syrups, and liquors) and escapes to the atmosphere through the carbonation vents.

Wastes from Heaters, Evaporators, and Pans

Air emissions from heaters, evaporators, and pans are limited to noncondensable gases created and driven out of a solution by heating the in-process products. Ammonia has received the greatest attention, but volatile organic compounds (**VOCs**) are also included.

Cleaning heating surfaces creates waste boil-out solutions which include acids, soda ash, sodium hydroxide, and the salts they create. Water condensed from steam during these processes often contains ammonia and BOD.

Wastes from Sugar and By-product Storage

Handling, storing, and shipping of sugar and by-products create dust and scrap material. Dust control is necessary not only to reduce particulate emission but also to reduce explosion potential because these dusts are explosive. Spills of sugar and by-product have the potential to pollute

surface and groundwater and to generate odor. Under the right conditions, molasses can spontaneously decompose, which causes an **explosion** if it occurs quickly or generates heat and carbonizes if it occurs slowly.

A response plan greatly reduces the likelihood that a **product spill** will cause environmental contamination. Storm-water runoff permitting requires facility managers to anticipate problems and put in place appropriate practices.

Wastes from Steam and Power Production

Beet-sugar facilities produce steam and electricity with coal, oil, and natural gas boilers. Air pollutants generated from combustion include sulfur dioxide (SO_2), oxides of nitrogen (NO, and NO_2, collectively called NO_X), unburned fuel or VOCs, carbon monoxide (CO), carbon dioxide (CO_2), air toxics (e.g., mercury, hydrochloric acid, and dioxins), and **particulate matter** (PM, PM_{10}, and $PM_{2.5}$). **Opacity** is the haziness or lack of transparency of a stack emission caused by particulate emissions scattering light.

Boiler ash is the most prominent solid waste. Pollution control wastes can be significant as well. Particulate matter, SO_2, and air toxics are removed from the stack gases often are transferred to the ash or water. Facility operators may have to treat these media or dispose of them appropriately.

Wastes from Process Chemicals and Fuels

Process chemicals, fuel leaks, and spills have the potential of damaging the environment. Some chemicals, such as SO_2, HCl, soda ash, and gasoline, are basically hazardous.

When considering the use of a chemical in a plant, the food safety, general safety, and environmental issues for that chemical should be investigated thoroughly. All chemical suppliers provide **material safety data sheets** (MSDS) with this information. Facilities managers should develop plans for cleaning up spills of each chemical used.

If a facility stores petroleum products in excess of 10000 gallons and a spill can reach waters of the United States, the EPA requires the facility to develop a Spill Control and Countermeasure plan.

Other Wastes

Beet-sugar facilities generate other solid waste that needs attention, including trash and garbage, laboratory wastes, used oils, parts-cleaning fluids, batteries, fluorescent lights, computers and electronics, asbestos, lead paint, other hazardous wastes, PCP-containing fluids, chlorofluorocarbons (CFC), scrap metal, sludge from wastewater treatment, oily rags, and construction scrap.

AIR POLLUTANTS

Air pollutants are any chemical, physical (e.g. particulate matter), or biological agent that has detrimental effects on the natural characteristics of the atmosphere (air above Earth's surface). The three general categories of air pollutants—gases, particulate matter (including opacity), and odor—are not independent, but they are usually treated independently by regulatory authorities.

As mentioned earlier, air pollutants from fuel combustion include SO_2, NO_X, VOCs, CO, CO_2, air toxics, PM, and opacity. Combustion is not the only source of these compounds, but it is the most significant. Other air pollutants produced at beet-sugar factories include ammonia (NH_3), chlorofluorocarbons (CFCs), and hydrogen sulfide (H_2S). NH_3 is released from nonsugars during the purification of beet juice. Some of the ammonia is released directly to the atmosphere by noncondensable gas venting. The remaining NH_3 is carried into various water streams. CFCs are low-boiling-point compounds (e.g., $CFCl_3$ that is used as refrigerant) that destruct ozone layer, known as ozone-depleting compounds. The ozone layer filters harmful rays from sunlight preventing damage to us. H_2S is highly toxic and generally associated with wastewater systems, though it can come from other sources as well.

Besides fuel combustion, other sources of VOCs include juice purification, pulp drying, noncondensable gas venting, pond emissions, and wastewater treatment. Volatile organic compounds vaporize at pressures and temperatures commonly used in sugar manufacturing. Dust is generated by traffic, the handling of raw materials and solid wastes such as coal and limestone, precipitated calcium carbonate, dirt, and ash. Ponds, wastewater treatment systems, dirt and mud handling, and pulp drying are sometimes sources of problematic odors. VOCs and H_2S emissions cause most of the environmental odors at beet-sugar processing facilities.

Although ammonia is not highly regulated, its ability to combine with atmospheric SO_2 to form ammonium sulfate particles is raising concern and could be regulated soon. In Europe, **ammonia emissions** are receiving regulatory attention because they can contribute to acidification of soils. Therefore, some factories install **ammonia-stripping** equipment to remove ammonia from condensate. Ammonia is removed by raising the pH of the condensate to about 10.5 (by adding caustic), followed by bubbling air through it.

Air pollution control strategies are usually some combination of prevention, treatment, and controlled release. The exact combination selected for a facility depends on regulation and economics.

Beet-sugar producers prevent air pollution by increasing energy efficiency, using clean fuels, using clean-burn technologies, and burning biofuels. Increasing energy efficiency throughout the factory reduces fuel consumption. Burning clean fuels (fuels low in sulfur, nitrogen, sodium, and heavy metal content) reduces the pollution created by these elements. Clean-burning technologies prevent the creation of pollutants during the combustion process. The use of biofuels uses energy from the sun, captured and stored by living organisms. Carbon dioxide emissions from these are part of the natural carbon cycle.

Most problematic odor from beet-sugar manufacturing comes from wastewater, mud, or lime waste. Minimizing or eliminating these are key to preventing odor. In addition, the correct design and operation of wastewater ponds and treatment systems keep them from emitting odorous gases.

Neighbors sometime complain about the odors associated with direct-fire beet-pulp dryers. Because this is an unavoidable by-product of this type of dryer, beet-sugar producers can do little to prevent the odor. Shutting the dryer down and selling wet pulp or replacing the dryer with a steam beet-pulp dryer is sometimes possible. However, wet pulp can have its own odor problems.

The removal of air pollutants from gas streams can be effective and is often the only economically viable option. Cyclones, filters, wet scrubbers, and electrostatic precipitators will remove particulates. Chemical agents can be introduced in the combustion chamber or to the

flue gases to react with the gaseous pollutants and keep them in the ash or to convert them to inert (not harmful) compounds.

End-of-pipe strategies can generate problematic waste streams that facility personnel have to address. Any evaluation of end-of-pipe strategies must examine the costs associated with safely handling and disposing of these waste streams.

The environment does have some capacity to handle air pollution, but it is clear that the current level of atmospheric emissions is too much for the natural environment. The ill effects of **acid rain (**see Glossary), photochemical smog, greenhouse gases (see Glossary), atmospheric mercury, and doxin are well documented. There is a widespread desire to reduce the atmospheric pollutants, but it is a slow business.

Controlling Sulfur Emissions

Burning sulfur-containing fuels creates sulfur (SO_2). Sulfur is required by plants and animals, so both fossil fuels and biomass (raw material from plants and animals) contain it. The amount of sulfur in fuels varies, depending on the source and form. Natural gas purchased commercially from large utilities generally is low in sulfur. (In some areas, natural gas contains high sulfur.) The sulfur content of fuel oil, coal, and biomass varies with the source.

Sulfur dioxide is a **toxic** gas that can kill people outright at 500 parts per million (ppm). There are several documented incidents of hazardous elevated atmospheric SO_2 from human activity. In the Meuse River Valley, Belgium, December 1938, and in London, England, December 1952, there were two incidents of elevated SO_2 that caused many deaths. Besides harming people, SO_2 can kill or otherwise harm animals and plants.

Acid rain is probably the most publicized and important consequence of SO_2 emissions. In the atmosphere, SO_2 combines with water and oxygen to form sulfuric acid. Acid precipitation landing on poorly buffered soil and water will acidify it, changing its ability to sustain life. Methods of controlling SO_2 emissions include the use of low-sulfur fuels, fluidized bed coal combustion with powdered limestone, and flue gas desulfurization.

Choosing a low sulfur fuel reduces the amount of SO_2 generated during combustion. Any fuel, natural gas, biologically derived fuel, fuel oil, or coal can be low in sulfur. Sometimes fuels are naturally low in sulfur, and sometimes fuels are treated to remove the sulfur.

Lime ($CaCO_3$) mixed with coal in a fluidized bed combustion system causes the SO_2 generated by combustion to react with the calcium oxide (CaO) formed from the lime. This reaction forms calcium sulfite ($CaSO_3$), a solid that remains with bottom ash or is removed from the flue gas with the fly ash. Coal and $CaCO_3$ need to be properly sized and added at the correct ratio.

In flue-gas wet scrubbing systems, water reacts with SO_2, forming aqueous sulfurous acid. Often, wet-scrubbing systems designed to remove SO_2 use calcium hydroxide to neutralize the acid. In these systems $CaSO_3$ can cause scale on pipes, pumps, and pits. Operators need to monitor flows and the pH, making sure that the scale does not plug the system. Too much scale buildup necessitates shutting down the system for de-scaling with chemical or mechanical methods.

After environmental engineers establish proper water flow, gas flow, and pH values for the wet scrubber, they need to develop and communicate the correct operating parameters. Operators need to understand that operating the pollution control equipment is required. In addition, the engineers must develop and operations personnel must use record-keeping systems that meet all regulatory requirements.

Scrubber equipment requiring maintenance includes pumps, valves, pipes, meters, and controls. An effective **particulate matter** (PM) **program** should be established for all components. Maintenance and engineering personnel must establish emergency maintenance procedures to ensure that all permit requirements are followed for the shutdown of pollution control equipment.

Electrostatic precipitators use electrically charged plates to attract particulates. Introducing powdered calcium hydroxide [hydrated lime, $Ca(OH)_2$] or powdered CaO prior to the precipitator causes SO_2 to react with lime to precipitate $CaSO_4$, which will be removed by the precipitator. It is important to add sufficient, appropriately conditioned lime. Operating parameters for electrostatic precipitators are covered in the upcoming Particulate Matter section.

Controlling NO_X

For simplicity in the environmental literature, NO_X refers to oxides of nitrogen (NO and NO_2), which are hazardous. During combustion, the high temperature provides the energy to change nitrogen (N_2) and oxygen (O_2) into nitric oxide (NO):

$$N_2 + O_2 + \text{Heat} \rightarrow 2NO$$

In addition, the nitrogen contained in fuel will form NO upon burning. Once in the atmosphere, NO reacts with air to form nitrogen dioxide (NO_2), a reddish-brown gas.

If breathed, NO is absorbed by the blood and attaches to hemoglobin, reducing oxygen uptake. NO_2 at 500 ppm will cause death within two to four days. Exposure to 150 ppm will cause death from bronchiolites obliterans (a lung disease) in three to five weeks. An exposure for an hour to 50 ppm will cause inflammation of lung tissue for six to eight weeks (Manahan 1994).

In addition to these health effects, NO_X harms the environment in a number of ways:

- Contributes to photochemical smog formation
- Forms nitric acid (HNO_3) in the atmosphere, which contributes to acid rain
- Reacts with particles and other airborn chemicals to form toxic chemicals
- Provides excess nitrogen to water bodies, contributing to eutrophication.
- Contributes to **global warming** (temperture increase on the Earth in recent decades)
- Harms plants and animals directly and through the chemicals mentioned above

Strategies for controlling NO_X revolve around the fact that its formation is favored by high-temperature and high-excess oxygen. By reducing the combustion temperature, and reducing excess air during combustion, or both, boiler operators can control NO formation. Once created, NO can be removed by adding ammonia (NH_3) to the hot flue gases.

Lowering the combustion temperature reduces the amount of NO formed for a given volume of fuel burned. This is usually accomplished by introducing cool air, or a cool inert gas, into the combustion chamber. Unfortunately, reducing the temperature also reduces the amount of useful energy available from the combustion. Controlling NO in this way reduces fuel efficiency.

Reducing excess oxygen not only reduces the amount of NO but also increases carbon monoxide emissions. To solve this problem, boiler manufactures have made two-stage burners. In the first stage, the fire is hot, but airflow is reduced to a minimum. In the second stage, cool excess air is added to complete the combustion of CO created in the first stage. Neither stage has both conditions necessary for NO production: excess oxygen and high temperature.

A similar strategy is used in coal-fired boilers. By reducing the forced draft to a minimum, operators reduce the excess oxygen moving through the hottest part of the boiler, which is the burning coal bed. Then, by increasing the over-fire air, they can burn away the excess CO. Modern boilers are designed to operate this way, but older boilers can be modified. Operators must always be careful not to reduce the forced draft so much that the grate gets damaged by trapped heat.

Boiler operators need to understand the methods employed for controlling NO. Operators educated in NO controls can spot trouble, make the necessary adjustments, keep parameters within the required limits, and call for help when necessary.

Controlling Carbon Monoxide

Carbon monoxide (CO) is colorless, odorless, and deadly. It binds tightly with hemoglobin, preventing oxygen transport from the lungs to the rest of the body. Incomplete combustion of fuel, caused by low oxygen, creates CO. Sources of CO include boilers, limekilns, direct-fired dryers, and internal combustion engines.

Having adequate oxygen concentration in the firebox ensures lower CO. Sometimes operators can accomplish this through meters and controls. Other times, the design or capacity of the air-moving equipment is inadequate and must be upgraded. Balancing CO and NO control with excess air was discussed in the preceding section.

Carbon monoxide is emitted from direct-fired dryers when they are pushed beyond their design capacity. At Minn-Dak Farmers Cooperative, CO is a function of the dryer-drum inlet temperature. At 600°C, the CO emitted is relatively low. However, as the temperature increases, the CO increases in a nonlinear fashion, reaching more than 400 ppm at just over 900°C. Charring the beet pulp during the drying process creates CO. Higher inlet temperatures increase charring and CO production.

To control CO from internal combustion engines, the engines must be properly tuned and the emission control devices, such as catalytic converters, must be operating correctly.

Controlling Volatile Organic Compounds

Volatile organic compounds (VOC) are carbon and hydrogen compounds that often contain oxygen, sulfur, nitrogen, fluorine, and chlorine as well. VOCs are produced and emitted by plants, animals, and microorganisms. VOCs also result from industrial processes, internal combustion engines, and a wide variety of other human activity.

The most publicized effect of VOCs is their role in producing photochemical smog. According to the EPA, about 58% of the VOCs are due to industrial processes. The rest come from internal combustion engines, aerosol propellants, and vehicles used to apply paints and coatings. Stagnant air, NO, VOCs, and sunlight are required to produce photochemical smog. The major sources of VOCs in beet factories are direct-fired pulp dryers, boilers, wastewater systems, and decomposing beets and pulp.

Controlling VOCs from pulp drying is difficult. Developing a market for wet pulp or installing a steam-powered pulp dryer are two methods that have been employed. The storage and handling of wet pulp can have associated odors and VOC emissions. A steam dryer requires a significant capital outlay but has the advantage of reducing energy consumption.

Controlling Particulate Matter

Particulate matters (PM) are small particles of suspended solids in the air. In a beet factory, PM come from boilers, pulp dryers, pellet drying and handling, sugar drying and handling, and fugitive sources. These sources are usually regulated through a facility's Title V Permit to Operate. Permits usually specify annual, daily, and hourly limits.

Particulate matter is classified by its size (Dust, PM_{10}, and $PM_{2.5}$). Dust particles are relatively large and pose limited environmental risk. Environmental engineers have classified as particulate matter particles smaller than 10 and 2.5 microns as PM_{10} and $PM_{2.5}$, respectively.

The smaller the PM, the more hazardous it is to humans and animals. When nonsoluble PM_{10} is inhaled, it becomes permanently embedded in the lungs, causing irritation and possible disease. $PM_{2.5}$ is so small that it can pass directly from the lungs to the blood stream, which transports any hazardous materials it contains to all parts of the body.

Fugitive dust is the PM created by traffic or from wind picking up waste lime, dirt, and other collected materials. The application of water or calcium chloride to gravel roads temporarily reduces dust. Paving problematic roads is a more permanent solution. Not letting dusty materials accumulate on site or making sure storage areas are protected from the wind also reduces fugitive dust problems.

Fossil and biofuels may or may not have particulate emissions. Gases such as natural gas, liquid petroleum gas, and biogas burn without emitting particulate matter. Coal and biomass combustion creates PM. For these sources, end-of-pipe strategies such as cyclones, electrostatic precipitators, packinghouse dust collectors, and wet scrubbers are usually used. Each of these technologies works well within its design criteria.

Cyclones or multiclones are designed to spin flue gases through an outer tube, and then reverse the direction through an inner tube. Particles in the gas are forced to the outside of the outer tube, slow down because of friction, and then drop to the bottom and are removed. The change in direction of the gas helps prevent re-entrainment of the particles.

Cyclones and multiclones are designed to work within specific airflows and pressure drops. Operators need to be aware of the critical values for their particular devices to ensure that they are operating correctly.

To maintain proper airflow, the inlet channels for cyclones must remain clean. If some of the cyclones become plugged in multiclones, the remaining cyclones will not function properly because the velocity of the air through the open cyclones will be higher—carrying particulates through the bank. Personnel must periodically inspect cyclones and multiclones for plugging.

Fabric filters or packinghouse dust collectors work well to remove particulates. The filter material depends on the temperature and airflow through the filter. Higher temperature materials such as fiberglass and polyaramids (e.g., Nomex) work well but are expensive. A wide variety of materials useful for filtering streams at ambient (surrounding) temperature are available.

In time, filters become plugged with particles, making a cleaning cycle necessary. Some designs incorporate a blast of air in reverse of the normal airflow. Others physically shake the filter. Some designs isolate the filter from the airflow during the cleaning cycle and some do not.

Personnel must take care to install filters properly to avoid leaks. Leaks are detected visually or through the use of optical, electronic, and mechanical monitors. Personnel need to respond quickly to leaks.

Fabric filters are vulnerable to fires, especially in pulp dryer and coal-fired boiler applications. Monitoring the gas inlet and outlet temperatures will allow the quick detection of fire. Whenever workers are going to open packinghouse dust collectors for maintenance, they need to check the temperature gauges and feel the outside of the collector before opening it. If it is hotter than normal, suspect a fire. Opening a packinghouse collector may supply a lot of oxygen to a smoldering fire and thus be dangerous. Other safety features necessary for packinghouse collectors include explosion venting and steam-out sprinklers or water sprinklers.

Condensation is often a problem for packinghouse dust collectors used in dryer or cooler applications. Water will condense on the filters and walls of the packinghouse (baghouse), wetting particulates and causing the dust collector to plug. For high-moisture streams, the air temperature must be higher than the dew point of the air. Insulating dust collectors and ductwork helps in these applications. Operators must monitor the collector inlet and outlet temperatures and make necessary corrections to keep the air stream above the dew point.

As particles move through air, they acquire an electrostatic charge. As these charged particles move through an electric field, they are subjected to a force that is proportional to the strength of the electric field and the charge. This is the phenomenon that highly efficient electrostatic precipitators use. Gas streams with particulates pass between charged plates. The electric field forces the charged particles to the plate, where they lose their momentum and are collected.

Wet scrubbers use water to remove PM. If a particle-laden gas makes sufficient contact with water, the particles will pass into the water, leaving the gas clean. Wet scrubbers are designed to maximize the contact between flue gas and water. In the Venturi design, the gas is forced through a constricted pipe where the scrubbing water is injected at right angles to the gas flow. The water breaks into small droplets, which remove small particles from the gas. Another design uses water sprayed into the gas stream, and still another uses the surface of a medium to increase the water–gas contact. Critical parameters are water and gas flow rates, and differential pressures in the scrubber. The smaller the particle that needs removal, the more difficult it is to remove.

Controlling Ammonia

Ammonia is considered a toxic air pollutant, a precursor to atmospheric particulates, and a contributor to the acidification of soils. Ammonia is emitted from juice purification, from the noncondensable vents from evaporators, heaters, and pans, and from wastewater ponds and treatment systems.

The nitrogen-containing nonsugars in sugarbeets are the primary source of ammonia emitted by beet-sugar facilities. The ammonia is produced in juice purification, and purged from juice at a high temperature and pH in evaporators. Modifying growing practices to increase sugarbeet purity reduces the amino content of the beets and, thus, ammonia emissions.

Ammonium sulfite used as a sulfite source by some facilities is another source of ammonia. By finding a different sulfite source, these emissions facilities can further reduce emissions.

Some beet-sugar manufactures collect the noncondensable gases and inject them into wash water or wastewater, where the ammonia becomes trapped. This does not eliminate the problem; it just transfers to another media.

Ammonia emissions from wastewater treatment can become significant if the **nitrifying bacteria** (the bacteria that convert ammonia to nitrate in aerobic systems) are lost. Nitrification

bacteria require about 10 days to grow, so wastewater-treatment system operators sometimes have to slow down the flow of wastewater through the system to allow the bacteria to recover.

Controlling Greenhouse Gases

Greenhouse gases are those **anthropogenic emissions** (emissions caused by humans) that through their accumulation in the atmosphere are increasingly trapping heat and affecting the earth. Greenhouse gases associated with beet-sugar manufacturing include carbon dioxide, oxides of nitrogen, ammonia, and methane.

Reducing fossil fuel combustion is one of the best strategies for reducing greenhouse gas emission. It reduces both CO_2 and NO_X. In addition, reducing steam generation, which is one of the greatest expenses for beet sugar manufacture, can have a significant payback.

When considering new projects, factory engineers and managers need to consider the total environmental cost and future regulatory requirements. Regulations dealing with greenhouse gas generation are in their infancy. Any capital project analysis needs to include expenses associated with greenhouse gas emission.

NOTE

Carbon dioxide and water have not been considered pollutants, but both absorb infrared radiation (heat) released by the earth. They act like a one-way filter, like the glass in a greenhouse, allowing the sun's rays to enter the atmosphere but preventing the infrared radiation (longer wavelength) to leave the earth. This is called the greenhouse effect. The name is from the similar effect that a greenhouse utilizes to increase plant growth.

LIQUID WASTES

Some **liquid wastes** produced during beet operations are environmentally pollutant and some are not. As mentioned previously, water has physical, chemical, and biological components. A variety of tactics are used to ensure that water discharged from the processing facility does not harm the environment. Governmental regulations require that water discharge meet both physical and chemical quality parameters.

Physical water quality parameters include temperature, color, and turbidity. Each aquatic organism has specific temperature requirements. Changes in water temperature will alter which species thrive or even survive. Water temperature affects aquatic organisms indirectly as well by changing the dissolved oxygen concentration. Color reduces the penetration of light into water, reducing photosynthesis and upsetting natural balances. Colored discharges have an unpleasant appearance and often indicate incomplete treatment of the water.

Loss of water clarity is an indication of suspended solids. Like color, suspended solids can reduce the penetration of light. The total suspended solids (**TSS**) concentration is measured by filtering water through a fine filter and then weighing the solid retained on the filter (see Chapter 11). Filters with smaller pores will catch more particles, so operators need to consider this when comparing data from different tests.

Chemical water quality parameters include organic and inorganic dissolved solids as dissolved gases. Dissolved organic compounds (e.g., sugar) when associated with nitrogen and

phosphorous provide microorganisms with food. Too much of such organic matter can cause explosive microorganism growth that will lower the dissolved oxygen content of a water body. In extreme cases, microorganisms can use up all the oxygen and create an **anaerobic** (without oxygen) condition. Altering the oxygen content of water will change the type of organisms that can thrive or survive.

The amount of organic matter in water is gauged by its biochemical oxygen demand (**BOD**), chemical oxygen demand (**COD**), and total organic carbon (**TOC**). These three parameters measure slightly different properties of the wastewater, but any one can be used for monitoring wastewater system performance because they correlate well to each other.

BOD is the amount of oxygen required by organisms to consume dissolved organic matter. Aqueous aerobic microorganisms (those requiring oxygen to live) use up about 1.1 g of dissolved oxygen (**DO**) for 1 g of sugar consumed. The standard test is run for five days and labeled BOD_5. Although microorganisms will not consume all the organic matter in five days, the amount of oxygen consumed is proportional to total oxygen needed for the microorganism to use all organic matter.

COD is the amount of oxygen necessary for a chemical oxidizing agent to oxidize all the reduced species (including organic matter) in a given amount of wastewater. COD will usually be greater then BOD. The chemical oxidizing agents used in this test oxidizes all organic species consumed by microbes in the BOD test, as well as organic and inorganic chemicals that microorganisms cannot consume.

TOC is the amount of organic carbon in a given amount of wastewater. This will be lower than either BOD or COD. For example, only 0.43 g of carbon are in 1 g of sugar.

Pesticides and **herbicides** are biologically active organic and inorganic compounds that can have detrimental environmental effects. Other inorganic compounds that can affect water quality include acids, bases, sulfur, and heavy metals.

In addition to dissolved oxygen, other dissolved gases include ammonia, carbon dioxide, hydrogen sulfide, and methane. These are all either organic or inorganic, but they are treated differently than dissolved solid compounds because dissolved gases have special properties.

Note: The analytical procedure for BOD and COD are given in Section 9 of Chapter 11. For more information, the author recommends using analytical methods from the *Standard Methods of Water and Wastewater Analysis* published by the American Public Health Association Publications.

Reducing the use of water and preventing its contamination is often an economical way to reduce water pollution. The benefits of this practice include reducing the capital investment in, and operating costs, of wastewater treatment. In addition, it reduces the likelihood that wastewater will cause environmental problems such as odors, groundwater contamination, and surface water contamination.

Proper operation and maintenance of harvesting and piling equipment reduces the dirt and weeds brought to the factory, which reduces the volume of water required to clean the beets. Replacing wet flumes and beet washers with dry handling and cleaning systems can reduce the amount of wastewater generated as well as the sugar lost to these systems. Well-maintained and operated washhouse equipment increases wash water reuse. Controlling bacteria in flume water through pH or antimicrobial compounds keeps wash water free of acids and other waste products. This prevents equipment corrosion and keeps to a minimum the blow down from these systems to wastewater treatment.

The use of dry air-pollution control methods instead of wet-scrubbing techniques prevents water contamination from these sources. Most water used to clean air streams requires treatment before being discharged.

Separating solids from water as quickly as possible reduces water contamination. Prolonged contact between solids and water promotes microbial degradation of the solids and dissolution of both organic and inorganic compounds. Pressing or decanting mud from wash water, pressing precipitated calcium carbonate from purification, and pressing wastewater mud (sludge) are examples of this prevention technique.

Water generated by condensing vapors from the evaporated juice always contains small amounts of volatile compounds from the beet juices. However, poorly maintained heat exchangers can leak juice into condensate and greatly increase the treatment required for this water. Monitoring condensate waters for sugar traces and quickly repairing leaks when they are found reduces the amount of contamination in the condensates.

The following structural and operational controls will reduce contamination of storm water runoff:

- Monitoring beet storage piles for spoiling beets and quickly removing them once detected
- Keeping storage areas dry by constructing the facility away from low areas
- Building berms around storage areas to prevent storm water run-on
- Building dikes and catch basins around a piling site prevent, contaminated runoff from reaching streams and lakes

Reducing wastewater and taking steps to reduce water pollution often has higher upfront costs, but usually reduces operating costs as well as the risk of polluting air and water off site.

WASTEWATER TREATMENT

Wastewater treatment is often divided into three categories:

- Primary
- Secondary
- Tertiary

These are not completely independent categories because more than one type of treatment can occur in a single structure:

Primary treatments remove dirt, trash, and other insoluble debris, usually by settling and screening wastewater.

Secondary treatments remove sugar and other dissolved organic materials. Secondary treatments usually use microbes in ponds, basins, or tanks to remove organic material by feeding on it and converting it to carbon dioxide, methane, and sludge.

Tertiary treatments remove nutrients such as nitrogen and phosphorous, and toxins such as pesticides and heavy metals. Some tertiary treatments are biological, but most are a combination of chemical and physical methods.

All these methods have their own waste products that need treatment and disposal, further increasing the cost of wastewater treatment.

Beet-sugar factories treat their wastewater in the following ways:

- Treatment in ponds: Used by all factories
- Active treatment: Used by all factories

- Anaerobic treatment: Used by some factories
- Aerobic treatment: Used by some factories
- Denitrification treatment: Used by some factories
- Land application: Used by some factories

Aerobic (requiring air) and anaerobic treatments are biological process for separating organic compounds from wastewater. The bacteria in aerobic systems use oxygen (O_2) for their reactions. Bacteria in anaerobic systems use oxidized elements (such as sulfur and nitrogen) of the organic compounds for their reactions. **Anaerobic systems** (also known as **activated sludge system** or Anamet) are closed systems, thus creating less odor emissions.

When comparisons aerobic systems and anaerobic systems, the aerobic bacteria do much better at using up the available chemical energy than anaerobic bacteria. The products of aerobic digestion are carbon dioxide (CO_2), nitrate (NO_3^{-1}), sulfate (SO_4^{-2}), and water; they are much lower in energy than the anaerobic products of methane (CH_4), hydrogen sulfide (H_2S) and ammonia (NH_3). Thus, the chemicals released from anaerobic systems contain high energy.

Gases emitted from properly designed and operated aerobic systems are not odorous, toxic, or flammable. Those emitted from anaerobic treatment are both toxic and flammable. Employees working with anaerobic systems must be trained to handle the hazards. In addition, the facility must install proper safety devices such as gas monitors and flame arrestors.

Anaerobic systems must be designed and operated to safely control these gaseous emissions, which is usually through burning the anaerobic gases. The anaerobic gas, or biogas, often replaces purchased fuel in boilers and pulp dryers.

Both aerobic and anaerobic systems generate solids known as **sludge**, or biosolids. The particles of sludge are composed of bacteria cells as well as inorganic matter such as dirt and calcium carbonate. Aerobic systems generate more biosolids for a given amount of organic load treated because they utilize the chemical energy better.

Types of Ponds

Ponds can perform four separate functions: sedimentation (settling), equalization, microbial treatment, and holding (see Figure 10.3). A pond will perform from one to all functions, depending on its design and operation. Different ponds are explained next.

Settling Ponds

Most suspended solids, such as dirt, weeds, and beet pieces can settle to the bottom of a **settling** (MUD) pond given time. Beet-washing systems are designed to prevent solids from settling in pipes and vessels by keeping the water moving. Once in a pond the water movement stops, allowing the denser solids to settle to the bottom. After the solids have settled, factory personnel can drain or pump away the clarified water, being careful not to disturb the settled solids. The water is treated before reuse or discharge.

Equalizing Ponds

An **equalizing pond** creates a well-mixed, slowly varying effluent from a variable influent. For example, wash water from washing frost-damaged beets will have higher BOD than wash water



Figure 10.3 Factory ponds (beyond storage tanks)

from washing undamaged beets. If the treatment system has been processing washhouse blow down from nondamaged beets, the bacteria population in the system may be too low to handle the high BOD blow down. An equalizing pond with several days of buffering capacity will slow down the rate that the treatment system feed, BOD changes. This will give the microorganism population more time to grow enough to handle the higher loading.

Equalizing ponds reduce the effect spent processing chemicals have on wastewater treatment systems by neutralization and dilution. Waste acids and base solutions used for chemical cleaning can neutralize each other in equalizing ponds. Many processing aids have antimicrobial properties that, in sufficient concentration, can harm the wastewater treatment bacteria. Water in ponds dilutes these chemicals, minimizing their detrimental effects on wastewater-treating microorganisms.

Operating an equalizing pond is fairly easy. The water volume must be kept high enough to accomplish the buffering. It is always a good idea to keep a spare area in an equalizing pond for wastewater system upsets. Upsets usually require the system operator to reduce the daily throughput of the treatment system, allowing it to equilibrate to the current conditions. The extra pond capacity gives some time for this process.

Treatment Ponds

Some factories use ponds for all their wastewater treatment. This includes pretreatment, aerobic, and anaerobic ponds. Treatment ponds need to be designed and operated for the desired functions. These ponds may be aerated or unaerated. The power to supply aerators in aerated ponds is high. Treatemt ponds generate sludge and may need periodic cleaning.

Pretreatment or pre-acidification ponds have sufficient residence time to allow the bacteria to convert sugar and other materials to organic acids. The acids are then easily digested by

other types of bacteria in the next step of the treatment system. Pretreatment ponds can be either aerobic or anaerobic. Operators monitor the amount of organic acid in the effluent to determine how the pond is functioning. The required performance of a pond will depend on the wastewater system following the pond. Operators must develop enough data for each system to determine if the system is operating correctly.

Treating wastewater in ponds requires monitoring of the physical, chemical, and biological parameters of the influent into the pond, the pond itself, and the effluent from the pond. If the following three questions are answered positively, a pond is performing well:

- Is the pond removing the total amount of organic loading it was designed to remove?
- Is the pond processing enough wastewater to keep ahead of wastewater generation?
- Does the pond effluent meet all required quality standards?

Aerobic treatment ponds are effective if the waste streams have an organic loading between 500 and 2000 BOD. The design of the pond and the use of **mechanical aerators** determine the concentrations of organic material the ponds can treat.

Some aerobic ponds are passive; they rely on the diffusion of oxygen from the pond surface to supply oxygen to the bacteria. Other aerobic ponds are equipped with active aeration devices such as stirrers or bubblers. **Aeration** improves the quality of pond water by removing volatile gases and volatile organic compounds (VOC) from wastewater. So, **aerated ponds** can treat more pounds of BOD in a given time than non-aerated ponds. Note that passive and active aerated ponds are designed differently.

The key operating parameters for aerobic ponds are organic loading and dissolved oxygen. If the loading becomes too high or the dissolved oxygen (DO) drops too far, the pond may not be able to sufficiently clean the wastewater. In addition, it may emit odorous organic compounds. If the DO drops to zero, the pond will turn anaerobic, totally changing the biology of the pond, and the quality of effluent water as well. Gases emitted from anaerobic ponds are not only odiferous, but also constitute environmental and health hazards in sufficient concentration.

Listed next are some parameters that wastewater system operators can vary to ensure proper control of an aerobic pond. Not every pond is designed with all these controls, so operators will have to use the tools at hand:

- Control the flow to a pond
- Control the strength of wastewater to a pond
- Control the level of the pond

Some systems have been designed to accept all the wastewater a facility generates without any controls. In that case, the wastewater treatment operator must keep the factory manager apprised of the pond's performance, so factory personnel can take measures to reduce wastewater loading and generation if the pond is not performing up to requirements.

Because of the noxious gases emitted by anaerobic ponds, they are usually covered and have biogas collection systems. Two important parameters in monitoring anaerobic pond performance are the amount of biogas production and its quality. Both parameters are excellent indicators of the health of the microbe population.

Both anaerobic and aerobic processes are temperature dependent. Treatment ponds work well in warm weather, but lose their effectiveness as the water temperature drops.

Holding Ponds

The primary purpose of some ponds is to hold water. The water could be untreated water before treatment or treated water before reuse or discharge. Biological processes and sedimentation will occur in holding ponds as in the other ponds. It is helpful if the effluent from treatment has residual organic or nitrogenous compounds that can be removed by biological action in the holding pond.

Holding ponds need to be large enough for their intended task. Discharging water into rivers and lakes is often restricted because of low flows, high temperatures, and low DO. In these cases, a beet-sugar manufacturing facility may need to hold the water over an extended period.

Pond Operation and Maintenance

If ponds are designed and operated correctly, they are low maintenance, making them attractive. However, ponds can be costly in time, money, and community goodwill if they are expected to perform functions for which they are not designed. Though inexpensive, ponds do not operate themselves.

Always know the water depth and freeboard in any pond. Never fill a pond beyond its engineered capacity because wave action can erode a dike and cause a break. Keep track of all flows into and out of all ponds. Conduct weekly inspections of all ponds, including physical measurements and a water balance. If the flows and ponds levels do not match, investigate immediately. If a leaking pond is suspected, contact your factory management or environmental engineer. Methods engineers can determine the rate at which a pond is leaking.

Keep pond banks and roads accessible all year. When inspecting ponds, look for signs of leaks such as wet spots on the outside of pond banks. Look for erosion from wave action. Examine the areas around inlets and outlets for damage or water leaks. Look for signs of burrowing animals, such as gophers, foxes, and badgers. If any are spotted be sure to notify your facility's pest control team so that the animals can be controlled and the damage repaired.

Always keep plants to a minimum on pond banks. Grasses and weeds prevent adequate inspections. Plants that die and fall into the pond, can plug pipes, pipe inlets, strainers, heat exchangers, and pumps. If herbicides are used to control plant growth, be sure to follow all regulations. Consult with your local county agent and licensed applicator to select and apply appropriate herbicides.

Clay-lined ponds should never sit dry. Leaving a few feet of water in the bottom prevents the liner from drying out and cracking. It also prevents plants from growing through the liner.

Overloaded ponds produce an offensive odor, which irritates neighbors and triggers visits from regulatory authorities. If discharging is affected, it could limit the wastewater production, and ultimately, factory operation. Constantly monitor factory wastewater streams to quickly catch and stop inadvertent loses of juices, syrups, or molasses into the sewer. Monitoring the washhouse screening equipment and making sure it is working well will reduce plant material washed into ponds. Preventing overload is much easier than trying to deal with it after it occurs.

When a pond is overloaded, some emergency steps that operators can try include odor-masking agents, the addition of engineered bacteria, the addition of trace nutrients, and the addition of chemical oxidizing agents. These are usually expensive options that may or may not work.

Many companies sell powders and/or aerosols that can be applied to ponds to mask odors. Most beet-sugar companies have had limited success with these as they are hard to apply, especially in windy conditions.

Some firms sell engineered bacteria or "super bugs" that work faster than native bacteria populations. Another avenue is to apply mixtures of trace elements to ponds, like a vitamin dose. This can speed up the bacteria's activity, so that the organic loading can be more quickly consumed. Neither method is instantaneous but can reduce the duration of the overloading.

The addition of chemical oxidizing agents such as hydrogen peroxide or potassium permanganate helps oxidize organic matter in an overloaded pond. This can be a quick fix for a one-time event but is expensive and not recommended for ongoing operations. There are safety issues in handling these chemicals, so all personnel handling them must be trained.

If overloading is due to an accidental, one-time event, these methods are usually acceptable to the community and regulatory authorities. If a pond is repeatedly overloaded, the company will have to find a permanent solution. This may be to install active wastewater treatment, segregate highly loaded wastewater, put a cover over the pond, or find a way to reduce the loading of the water. Neither the community nor the regulatory authority will tolerate repeated odor or discharge violations.

For ponds in which sedimentation takes place, periodic pond cleaning is required. Methods used include suction of mud and sludge and cleaning with backhoes, draglines, clamshells, or front-end loaders. When cleaning ponds, care must be taken not to damage pond liners and dikes. After each cleaning and before resuming the pond's use, a qualified engineer should inspect it, measure the liner on the bottom and sides, and certify the pond's integrity.

Active Treatment of Wastewater

Beet-sugar producers use a wide variety of active wastewater treatment systems. The particular configuration for a facility will depend on many things including the wastewater volume, the loading, the weather, and the local regulatory environment.

Wastewater coming directly from the facility or a pond is often screened to eliminate beet pulp and other vegetative materials. The wastewater might require heating or cooling before introduction into the system because biological treatment systems run best in narrow temperature ranges. Treatment systems usually run between 20 and 39°C, so low-grade heat sources are usually sufficient to heat incoming water.

Beet-sugar facilities commonly use **anaerobic** and **aerobic** systems. As with the ponds, anaerobic systems work well for heavily loaded wastes, and aerobic systems work well for lightly loaded wastes. In both types of systems, bacteria utilize organic material for food. As bacteria grow, they produce extra cellular polymers and use them to catch dirt, plant matter, and other bacteria. This material accumulates the sludge and creates a home for bacteria.

Filters are used for removing fine particles and algae from discharge waters. If dissolved solids need to be removed, reverse osmosis is usually employed, but other methods are available.

Anaerobic Treatment of Wastewater

For high-strength wastewaters, **anaerobic treatment** (also known as **activated sludge system** or Anamet) has a number of advantages over aerobic treatment. Anaerobic systems are smaller,

do not require as much energy, generate much less sludge, and produce biogas. However, these systems start up slowly, respond to changes slowly, are "upset" easily, and operate well only within narrow pH and temperature ranges. In addition, the biogas is intrinsically hazardous.

The two most-used anaerobic reactors are the mixed liquor tank and the upflow reactor. The mixed liquor tank creates a suspension of sludge throughout the tank to maximize the contact of the sludge with the wastewater. The upflow reactor uses a granular sludge suspended on a media in the tank.

A mixed liquor tank is equipped with some type of mixing device(s), typically stirrers or bubblers. To prevent an explosion hazard, the bubblers use biogas and not air. Wastewater is continuously fed into the tank, and an effluent continuously comes out of the tank. The effluent contains the sludge, which must be separated from the wastewater. Several clarifier designs are used, but they all work similarly. In the clarifier, the flow slows enough to allow the sludge to settle. The settled sludge is pumped off the bottom of the clarifier and back to the mixed tank. Excess sludge is removed from the system for disposal.

The key to operating a mixed-liquor tank is maintaining sufficient sludge in the tank. Anaerobic bacteria are not efficient in their use of organic food. It takes a lot of food to grow a small population of bacteria. If the bacteria are lost from the tank for some reason, replacing them will take a long time. Good clarifier operation is essential to good mixed-tank operations.

Operators must monitor the settling rate of the sludge to make sure it is meeting the design criteria of the system. If a problem arises, only a few solutions are available. The addition of chemical flocculating agents, usually cationic polymers, increases the settling rate. This can be a good tool if the anaerobic digester is operating well but the sludge is not settling in the clarifier. However, if the sludge is not settling because of an upset condition in the digester, slowing the system down may be the only way to recover.

Anaerobic bacteria are the prima donnas of the wastewater world. Anaerobic bacteria are sensitive to changes in loading, temperature, food type, and pH. If everything is not just right, they will stop working.

The key to good anaerobic system operation is to keep the microbes happy. Keep the temperature at 39°C, and the pH above 7. Make all changes slowly, to give the bacteria time to adapt. Most anaerobic systems use a preacidification tank or pond as the first step, followed by an anaerobic reactor. If the pH of the digester starts to drop, sodium hydroxide (NaOH) or some other base must be added to maintain the pH. In addition to pH, other chemical parameters to monitor include alkalinity, BOD, TOC, COD, volatile organic acid concentrations, alkalinity, biogas production, total suspended solids, and total volatile solids. Parameters must be monitored in the influent stream, the tank, and the clarifier underflow and effluent.

A fast up-flow reactor is a simpler design than a mixed liquor tank. The tank contains a mesh that traps a granular sludge. The granular sludge has a bacterial content sufficient to consume the organic material in the wastewater. Excess sludge is swept out of the tank as part of the effluent. This design removes the necessity for a clarifier.

Recovering quickly from an upset is important. Most beet-sugar facilities do not have excess wastewater storage capacity. If a wastewater treatment system is processing wastewater too slowly, the factory may have to slow down operation. In warm weather, ponds containing high-strength wastewater will turn anaerobic and produce odors. Because the public's tolerance

for odor is low, slowing down anaerobic systems is usually without public relation consequences. On the other hand, if the facility pushes wastewater through a system operating poorly, the processed water may be unfit for reuse or discharge.

A wide variety of conditions can cause an upset. Sometimes the only solution is to lower the system throughput enough to allow the system to recover. Operators can add nitrogen, phosphorous, trace elements, or other nutrients to promote fast bacterial growth. Pinpointing the cause of an upset can be difficult. Often, several actions are taken at once in hopes that one of them will work. Operators who use this method, however, never know which action or actions solved the problem.

Operators should try to discover problems as quickly as possible. By conducting tests daily, they can often pick up on problems just as they are starting. At Minn-Dak Farmers Cooperative, propanoic acid showing up in the anaerobic effluent is usually the first sign that there will be trouble in the tank. Wastewater personnel reduce the daily loading to let the bacteria population adjust to changing conditions. Another tactic is to increase the sludge concentration in the tank by adding flocculants or reduce the sludge wasting. In some cases, operators have started using caustic to counter a drop of pH. The earlier an upset is found, the faster it can be corrected and the less problem it will cause.

The biogas generated by an anaerobic system is between 60 and 80% methane (CH_4), with the balance mostly carbon dioxide (CO_2), water vapor, and small amounts of hydrogen sulfide (H_2S). It is explosive (when mixed with air), toxic, and corrosive. Anaerobic system design and operation must consider all these properties. Equipment enclosures accessible to people must have a gas warning system, and all parts of the system must be well maintained and functional at all times.

Factory and contract personnel need to understand the hazards of biogas and how to work with it. Personnel working in and around biogas systems must be trained in how to respond to biogas leaks, what actions they should take, and who they should contact.

Biogas handling systems often include blowers, pipelines, outside flares, burner heads inside boilers, and pulp dryers and flame arresters. Personnel working with this equipment need to know how each piece works, the potential hazards, and the actions they are allowed to take during normal and abnormal operating conditions. They must also know whom to contact when the work required is beyond what they are allowed to do.

Biogas from anaerobic digestion is an excellent fuel and helps pay for wastewater treatment. However, because each pound of sugar converted to biogas represents only a few cents worth of usable energy, it is a costly fuel. Keeping sugar out of the wastewater streams is more economical than converting it to biogas.

In summary, anaerobic systems are environmentally friendly but need skill and care in their operation. These systems clean organic matter from heavily loaded waste streams with little energy input. They produce much less sludge and are much smaller than aerobic systems. Anaerobic systems use a minimum of added chemicals and produce a biogas that can replace fossil fuels.

However, anaerobic systems are slow to bring up to speed. In addition, they become upset if they experience quick changes in feed constituents, strength, or temperature. The intrinsically hazardous nature of biogas puts equipment and personnel working with these systems at risk. Proper design, construction, safety equipment, and training are all essential. If designed, operated, and maintained correctly, anaerobic systems are an efficient way to treat high-strength wastewater.

Aerobic Treatment of Wastewater

Aerobic treatment works well for wastewaters less heavily loaded than wastewaters processed through anaerobic systems. Aerobic systems are often used if the facility does not have the space for ponds. Another advantage over ponds is that the facility can process the wastewater as it is produced even in colder weather when bacterial activity in ponds slows down.

Most aerobic systems consist of tanks or basins equipped with some type of mixing ability to suspend sludge and keep dissolved oxygen (DO) high enough. The power to supply mixing and aeration is high when compared to anaerobic systems. Aerobic systems separate sludge from the treated effluent with continuously operating clarifier or batch settling basins.

Unlike anaerobic systems, properly operating aerobic systems will have a inoffensive, earthy odor. The organisms expel low levels of CO_2, which mostly remain in solution as bicarbonate or precipitate in the sludge as calcium carbonate.

Monitoring aerobic system performance is similar to measuring anaerobic systems. Extra parameters required for monitoring aerobic systems are DO, NH_3, nitrite (NO_2), and nitrate (NO_3).

Like anaerobic systems, the key to successful operation is maintaining the proper bacteria population. Aerobic bacteria grow much faster than anaerobic bacteria and more fully utilize the energy in the food. As a result, they produce more sludge per given amount of BOD than anaerobic bacteria. As a consequence, aerobic systems start up faster and recover more quickly from upsets than anaerobic systems.

The excess sludge produced by an aerobic systems will accumulate much faster than with an anaerobic system. If the water is not heavily loaded, this may not be a problem. (Sludge removal and disposal are covered in the Solids Wastes section.)

Sludge age is a critical parameter for aerobic systems. To determine the sludge age, the operator must keep track of how much sludge is in the system and how much sludge is being removed each day. To calculate the sludge age, divide the amount in the system by the amount removed.

The best sludge age will depend on the needs of the system. If sludge is too old or too young, it will not settle well and cannot be maintained. If the system is expected to convert ammonia to nitrate, the sludge must be at least 10 to 20 days old because the nitrifying bacteria take longer to grow than other aerobic bacteria. The operator must always balance controlling sludge age with throughput and **BOD** removal.

There are a few ways to control sludge age. If a system uses a clarifier, the operator can increase the age of the sludge by increasing the depth of the sludge in the clarifier. Another method is to increase the concentration of the sludge in the basin. If sludge is not settling and too much sludge is escaping in the effluent, operators can reduce the throughput or add chemical coagulants and flocculants.

Denitrification Treatment of Wastewater

In some wastewaters, the removal of nitrogen compounds is necessary because their contents are high. Conversion of ammonia (NH_3) to nitrate ions $(NO_3)^-$ is an aerobic process called **nitrification**. The subsequent conversion of the nitrate to nitrogen gas (N_2) by denitrifying bacteria is an anaerobic process called **denitrification**.

Denitrification occurs both before and after aerobic treatment. First, wastewater is treated aerobically to remove BOD and to convert ammonia to nitrate. The effluent is then combined

with a slight excess of BOD in the non-aerated denitrification basin. The bacteria use up O_2 and the nitrate. As long as the operator carefully balances the BOD with the nitrate, the system efficiently converts the nitrate to nitrogen gas (N_2), which bubbles out of the water and is released into the air. If the operator adds too much BOD, the system turns entirely anaerobic. If the operator does not add enough BOD, the system only partially converts the nitrate.

During denitrification, the nitrogen is removed, and the BOD is consumed without additional aeration. Incorporating a denitrification basin not only removes nitrogen from the water but also lowers the energy relative to active aerobic treatment.

Maintenance of Wastewater Systems

A good maintenance program is essential to keep any wastewater system running. Preventive maintenance depends on both operators and mechanics. A good operator will know how every part of a system runs. System performance will often suffer when there is a mechanical problem such as a mixer, aerator, or pump malfunction. Operators need to use all their senses when making rounds. A noise, leak, vibration, smell, temperature difference, vapor, or anything else that is unusual should be investigated. Skilled operators will be able to diagnose many problems themselves and will either correct the problem or contact the appropriate maintenance personnel.

When problems are beyond the ability of the operators or the scope of their duties, they must rely on others to diagnose and correct the problem. Unfortunately, it is easy for plant maintenance personnel to put a low priority on wastewater treatment system maintenance because often it is not as pressing as the latest process crises. Wastewater operators must be persistent in bringing unsolved problems to the attention of the appropriate personnel. Unresolved problems can quickly escalate into breakdowns that threaten plant operations and regulatory requirements.

Wastewater treatment systems have a variety of maintenance issues. Problems with pipes include leaks, plugging, freezing, and scaling. Pumps, stirrers, and aerators fail due to mechanical, electrical, physical, and control problems. Pits will leak, fill up with solids, overflow, and emit toxic gases. Heat exchangers will leak between the hot and cold side as well as to the outside. They will scale up, blow gaskets, and plug. Screens will plug and scale up. Instruments and control systems will malfunction because of electrical or programming problems. Basins, tanks, and clarifiers will fill up with solids, develop leaks, and scale up. All metal part and plastic parts will corrode or erode.

Other parts of the preventive maintenance program should include scheduled lubrications and inspections. Diagnostic techniques include thermal scanning, oil analysis, vibration analysis, instrument calibration, and scheduled replacement.

As operators and maintenance personnel discover recurring maintenance issues, they need to bring them to the attention of plant management and engineering. Often, an examination of the system can spot the cause of the problem. Changes in materials, equipment types, and operations methods can reduce or eliminate maintenance problems.

Land Application of Wastewater

Applying untreated or partially treated wastewater to land both treats and disposes of wastewater. The bacteria in the soil will consume the BOD. Plants can use both the water and the nutrients. Some of the problems with land application include salt buildup in the soil,

oversaturation and pooling of applied water, contamination of groundwater and surface waters, mechanical issues with the application equipment, permitting from the appropriate regulatory authorities, and meeting all monitoring, record keeping, and reporting requirements.

Any land application should be done in close consultation with agronomists, soil specialists, hydrologists, and regulatory authorities. The agronomist can select the best type of crops to utilize nutrients. The soil specialist can determine the long-term implications for the soil. The hydrologist can assess the impact of the application on the ground and surface waters. The regulator can help with permitting requirements.

To use wastewater for land application, the salinity of the water must be appropriate for the soil type. Salt will cause some soils to harden on drying, making it difficult for germinating plants to punch through the surface. Soil and water tests can be used to determine how much salt a soil can take. Rotating the wastewater application with the application of other, less salty water can provide a solution if enough water is applied to the land to force the salt down into the subsoil so that it does not accumulate at the surface. Another solution is to segregate less salty wastewater streams that can be applied to land from salty ones that cannot.

When applying land water, operators usually have to do a number of analyses including ones for TDS (salt), nitrogen, phosphorous, and BOD. The total amount of water that an operator can apply depends on the concentrations of these constituents, the soil type, and the crop. Groundwater monitoring is often required, especially if local wells supply drinking water for farms or cities.

Discharge of Wastewater

To ensure that discharging does not interfere with the use of the water by people or alter the environment, governments regulate water discharges. Rivers and lakes have some capacity to accept water of different quality than their own without ill effects. Regulatory authorities are charged with the responsibility of determining what discharge water quality is not detrimental. By abiding with regulatory requirements, beet-sugar manufacturers can discharge water without adversely affecting the natural waters.

Usually, permits set limits on specific water quality parameters in the discharged water. In the United States, some limits are codified in the Code of Federal Regulations (**CFR**). To maintain local water quality, the limits for a particular factory may be more stringent than those listed in the CFR.

In addition, many permits require monitoring of the receiving waters and set limits on parameters within the receiving waters. As governments implement the Total Maximum Daily Loading regulations, receiving-water monitoring will become more important. These regulations require all facilities discharging within a given area to coordinate their discharges to ensure the natural capacity of a lake or river is not overwhelmed.

Permits require extensive record keeping and reporting. Records of discharge dates, times, durations, and volumes are usually required. Other required records include analytical parameters such as BOD, DO, TDS, and temperature, as well as laboratory certification and quality control. The permit usually spells out how long to hold the records.

The National Pollution Discharge Elimination System permits issued by the EPA or the designated state or tribal authority require monthly reporting. Personnel from the regulatory authority will usually make regular visits to a permitted factory to review the records, visually observe operations and discharges, and sometimes take samples.

If regulatory personnel take samples, be sure to have factory personnel split the sample for independent testing by a third-party certified laboratory. This can prevent a problem with an erroneous test result by the regulatory authority.

It is important for beet-sugar producers to obey the terms of the discharge permits. The permits not only protect the waters but also are legally binding. If a company violates the terms of the permits, they receive monetary penalties and regulator authorities can revoke the permit, taking away the company's ability to discharge, which can cause a temporary or permanent factory shutdown.

If regulators find that individuals have purposely violated permit requirements, they can bring criminal charges. If convicted, responsible individuals can face fines and jail sentences.

SOLID WASTES

Solid wastes of sugarbeet processing are operational wastes generated during operations. They are in either solid or semi-solid form.

Solid wastes of a beet factory can be divided into the following five groups:

- Disposable wastes (e.g., rocks, stones, and sand)
- Saleable wastes (e.g., carbonation-lime residue and dirt)
- Chemical wastes (e.g., used oil, laboratory, and process wastes)
- Construction wastes (e.g., used bricks from limekiln and scrap metals)
- Biodegradable wastes (e.g., spoiled beets, beet chips, beet pulp, and weeds)

Unlike air emissions and liquid wastes, solid wastes do not completely disperse into the environment. They do disperse to some degree but usually in an unacceptable fashion. Water will transport solids through dissolution or suspension, both of which generally have detrimental effects on ditches, rivers, lakes, and groundwater. If a solid waste is left exposed to air, it can generate dust or undesirable odors.

Solid waste generators must bear costs associated with treatemt, storage, transportation, and disposal. The following is a list of costs associated with solid wastes:

- Collecting the wastes
- Analysis of the wastes
- Temporary storage of the wastes
- Treatment of the wastes to make them fit for final disposal
- Transportation to the final disposal site
- Placement in the disposal site
- Postplacement care
- Record keeping
- Remediation
- Monitoring
- Permitting
- Legal cost
- Training

The four generic strategies for dealing with solid wastes are prevention, treatment for reuse or release, release into the environment, and storage. The generation of solid wastes triggers all associated costs no matter what method is used for disposal. Preventing the generation of a waste avoids all the associated costs and liabilities.

The two most important tools in prevention are better control of existing processes and selecting a different process. Management must identify the process targets, communicate the targets to employees, and verify that the employees are meeting the targets. Changing processes often requires significant capital and must meet corporate goals.

Wastes must often be treated before they are released to the environment or deposited in storage. Dewatering makes waste material easier to handle. Another method solidifies waste by cementing materials together. Such treatment can reduce potential environmental effects, reduce costs, or both.

Releasing solids to the environment must be done in a way that does not contaminate the soil, water, or air. Solid wastes not immediately released or never released are stored. Applying solid wastes to land is an environmentally friendly way for disposing of waste materials.

Before conducting any land application, be sure to consult local, state, and federal guidelines. One or more of these governing bodies will have rules regarding land applications, which can include the following:

- Solid waste type
- Operator certification
- End-user information
- Site suitability criteria
- Soil-testing requirements
- Notification requirements
- Site management, limitations, and restrictions
- Record-keeping and reporting requirements

Nutrients in solid wastes are both a hazard and a benefit. Leachate (leached out material from a semi-solid source) from solid wastes containing high concentrations of nutrients can pollute ground and surface water. If high-nutrient wastes are spread too thickly on agricultural land, the excessive nutrients can have toxic effects on growing plants.

The amount of nutrient-containing solid wastes applied to the land at a rate that supplies the appropriate amount of nutrients for an upcoming crop is the agronomic amount. A solid waste applied at an agronomic rate replaces other nutrient sources and poses no harm to the crop or the environment.

To determine the agronomic amount of solid waste to apply to a field, the nutrient content of the field, the nutrient content of the solid waste, and the nutrient requirement of the upcoming crop must be determined. The first two require sampling and chemical analysis. Because local conditions have a significant effect on nutrient requirements of the crops, local agricultural professionals should be consulted for this determination.

To prevent polluting, facility managers need to have waste-handling procedures in place. Everyone who works with solid wastes must understand and follow these procedures. Beet-sugar facilities generate a variety of solid wastes.

Handling Dirt, Rocks, and Weeds

Dirt, rocks, weeds, wood, metal, and trash brought to the factory along with the sugarbeets must be separated from the sugarbeets before processing. They create a significant solid waste problem. Because dry tare can range between 2 and 8% of the beet tonnage, 1000000 tons of beets processed will generate between 20000 and 80000 t of solid wastes for disposal. Dirt removed from the beets will be between 20 and 80% solids, depending on the method of cleaning and how it is dewatered. The water content of the waste material can significantly increase the cost of dirt disposal.

Although it is not possible to prevent these materials from coming into the factory, beet-sugar facilities can reduce their amount through policies that encourage sugarbeet growers to deliver clean beets and through operational practices at the piling stations. Economic incentives and education can encourage growers to purchase the right equipment as well as adopt operation and maintenance practices that reduce tare materials.

Piling operations offer another opportunity to reduce tare materials. Proper operations such as **beet drycleaning** (see Section 2 of Chapter 3) and slow operation can remove much of the dirt brought from the field. The dirt removed in the beet-drycleaning station does not need separation from the wash water. The dirt washed from beets must be prepared for disposal through separation from the wash water. This is accomplished in a number of ways, including the use of settling clarifiers, ponds, filter presses, and decanters.

Separation of Dirt from Wash Water

Because the factory must deal with the dirt and water, the technique used to separate them should be as complete as possible. The higher the amount of solids in the separated dirt, the less solid or semi-solid material has to be handled. Lower moisture dirt has less potential for odors and can reduce hauling and disposal costs. Low solids in the separated water are more usable than water high in solids. In addition, suspended solids in the clarified water will drop out in wastewater treatment ponds, head exchangers, pipes, and tanks, ultimately increasing wastewater treatment expenses. If the clarifier is operating well, the TSS in the clarifier will be below 1000 ppm (0.1%).

Solids Separation using Settling Clarifier

Settling clarifiers use the density difference between solids and water for separation. A clarifier is large enough to slow the flow of the water-dirt suspension, so the dirt settles faster than the upward fluid flow. By introducing the dirty water near the clarifier bottom, the thickened, settling mud helps catch the dirt from the introduced water. The thickened mud and clarified water are constantly removed from the clarifier, the thickened dirt from the bottom (underflow), and the water from the top (overflow).

The controlling parameters for clarifier operation include the TSS of the overflow, the density of the underflow, the amount of clarified water required, the capacity of the underflow-handling system, and the tolerance for bacterial growth in the clarifier. The clarifier operation will depend on the nature and amount of suspended and dissolved solids, pH, temperature, and required throughput of the feedwater.

Following are actions that operators can take to control the clarifier's performance:

- Change the character of the feed
- Reduce the throughput

- Increase the underflow relative to the overflow
- Change the temperature
- Change the pH
- Add chemical settling aids

The operator of a specific clarifier may not be able to adjust all of these because of equipment or other limitations. However, over time, facility managers can change equipment and operating procedures to allow for better operator control.

Settling is dependent on both the size of a particle and how it interacts with other particles. Bulk liquid flow and Brownian motion will cause small particles to bump into each other. Under the right conditions, the particles stick together, gradually becoming bigger and finally settling to the bottom. If this process is happening too slowly, the particles do not settle.

Because different types of soil settle differently, changing the character of the dirt can help settling. About the only way to change the character of the dirt in the wash water is to alter the source of the beets. This could be a good strategy if the amount of dirt coming in with the beets is too much for the clarifier to handle, or if washing too many spoiled beets has changed the settling characteristics of the dirt.

If it is not possible to alter the source of the beets, it may be necessary to reduce the amount of dirty wash water going through the clarifier. Increasing the retention time in the clarifier will increase the collisions a particle undergoes in the clarifier. This can be accomplished if there is a bypass around the clarifier to a settling pond. Slowing down beet receiving is an extreme measure but one that is sometimes unavoidable.

Increasing the volume of underflow relative to the overflow can increase the clarity of the overflow. Settled mud building up at the bottom of a clarifier reduces its capacity. Under these conditions, increasing the draw off the bottom removes the settled solids and restores the clarifier to its design capacity. If the dirt is just settling poorly, increasing the underflow will alter the bulk flow. This can help reduce TSS in the overflow, but the higher underflow volume will have lower TSS. Because the underflow is usually dewatered further, a lower suspended solids concentration can have consequences on that process. It can also increase sugar loss.

Increasing the temperature increases the collisions between particles in the clarifier and can help settling. On the downside, sugar will diffuse from the beets faster into warmer wash water. If this is tried, you should monitor the sugar content of the flume water and the total sugar loss into wastewater and mud.

Changing the pH of the water can affect settling because particles have different surface charge solutions with different pH values. By varying the pH, operators can find a pH that minimizes the surface charge, allowing the particles to stick together. Operators need to be mindful that keeping wash water pH between 7 and 10 controls corrosion and microorganisms, respectively.

Chemical settling aids are often useful in separating dirt from wash water. To select the right settling aid, you need to know why the particles are not settling. Because the particle type changes during a campaign, the type and amount of settling aid will also change. For example, a settling aid may work as long as TSS stays below some critical point, or the soil on the beets contains a minimum amount of humus. When these values are exceeded, operators may have to use a different settling aid or increase the system underflow to remove solids and reestablish the conditions under which the settling aid worked, or make some other change.

Many commercially available settling aids work with a variety of mechanisms. Anionic and cationic polymer will attract particles with oppositely charged surfaces. Increasing the calcium or aluminum content of the water can also overcome surface charges. Because of the wide variety of flume conditions and settling aid types, the choice of settling aid must be established experimentally and often.

Mud ponds with or without settling clarifiers are used to separate dirt from wash water. Mud ponds are designed so that water flow through them is slow enough to allow dirt and organic materials to settle. Allowed enough time, the uppermost water becomes clear and is removed for treatment.

After beet processing is finished, a thorough dewatering can take place. How much drying is desirable depends on the disposal method used. Excess water is pumped off and the drying power of sun and air are employed to maximize evaporation. The other aspects of mud pond operation and maintenance are similar to those of wastewater ponds.

Solid Separation using Mud Press

Pond and settling clarifier performance is often limited due to time, weather, and the character of the mud. The use of mud presses or decanters can dewater mud more quickly and to a much greater extent than settling clarifiers or mud ponds. Table 10.1 shows the different quantifies of water reduction from different levels of active dewatering. The water reduction allows for greater recycling in the washing and fluming systems, which helps reduce sugar loss and wastewater generation. In addition, the reduction in tonnage of mud can reduce the cost of hauling and spreading the mud.

The reductions shown in Table 10.1 are not without cost. Mud ponds and settling clarifiers operate with a minimum of operator interaction or maintenance, but mud presses have significant operation and maintenance costs. To coagulate and dewater the mud before pressing, properly prepared polymers must be added to the mud stream. Then the mixture must sit for several hours, during which, the polymers unwind (relax), increasing their effectiveness and lowering the cost.

The mud press operator must keep close watch on a number of parameters, including mud density, mud feed rate, belt alignment, polymer mixing and aging, and routine maintenance. The mud must be spread evenly across the press to prevent the belts from stretching and tearing. If the mud density varies too quickly, it is difficult to keep the polymer adjusted just right. If either too little or too much polymer is added, the mud will remain runny and not press.

Table 10.1 Reduction in Water and Mass by Solids Increased to 50% for 40 Dry Tons of Dirt (5% Tare on 1000t of Sugarbeets)

Initial Solids (%)	Initial Mud (tons)	Final Solids (%)	Final Mud (tons)	Water (%)	Mass Reduction (%)
15	267	50	80	82	70
20	200	50	80	75	60
25	160	50	80	67	50
30	133	50	80	57	40

The mud press belts need continuous cleaning. The cleaning water will have some organic loading that varies from 300 to 2000 BOD depending on the condition of the beets being washed. Over time the belts will clog and require chemical cleaning, especially if lime is used in the flume to control microbes.

Decanters are more forgiving than mud presses and more easily automated. They run better over a range of mud densities and do not require polymer. However, the dirt wears them out quickly, and they require significant routine maintenance.

Handling Carbonation Mud

Conventional lime-carbon-dioxide juice purification generates precipitate calcium carbonate, known as **carbonation mud**. Most factories thicken the mud with **filter presses** (plate and frame filters or membrane filter presses) to produce a cake-like substance, known as **carbonation-lime residue** (CLR, also known as **limecake**). Trucks haul lime residue from the factory to storage (either permanent or temporary). Carbonation-lime residue contains about 60% calcium carbonate, 30% water, and 10% inorganic and insoluble organic compounds (mostly beet-plant tissue). It also contains trace element, such as selenium, zinc, and molybdenum.

Plants that use rotary-drum filters, desweeten the cake with water and pump it to the lime pond. After the lime in the pond has settled, the water is decanted from the top and sent for wastewater treatment. The ponds are then cleaned with heavy equipment to make room for the next campaign's lime. Lime ponds are high in organic loading and have a great potential for polluting the environment, so most beet sugar producers have stopped using them.

If the right type of kiln is available, carbonation-lime residue can be reburned. The lime from the reburned carbonation-lime residue (CLR) has more impurities than lime generated by burning high-quality limestone, so some virgin limestone is often burned with CLR to increase its lime content.

The acid neutralizing power, trace elements content (it contains selenium, zinc, molybdenum, and more), and fine particle size make lime residue especially attractive as an agricultural soil supply. Lime application replenishes trace elements in the soil that have been depleted from years of cropping, significantly improving the soil fertility. The fine particle size allows it to be mixed well with the soil, reducing the amount required to neutralize acids and replenish nutrients and trace elements.

Lime residue can be used as an agricultural soil enhancer and pH adjuster. Agricultural demand for PCC is dependent on local soil needs. Some sugarbeet processing facilities sell PCC as a by-product, while others have no market and must pay to landfill it.

Handling Coal-Ash Waste

Bottom ash is usually inert with little potential for leaching out hazardous chemicals. Common practice is to dispose of bottom ash in landfills or use it as an aggregate for construction projects. Fly ash, however, has a number of associated hazards. It is easily blown by the wind and transported in storm water runoff, and it contains heavy metals that water can leach out. Unlike bottom ash, the method of storage must adequately protect fly ash from the elements.

Permanent disposal of fly ash must be into a landfill with adequate environmental safeguards. Characterizing the composition of the ash through elemental leachate analysis is required before disposal. The landfill operator requires a copy of the analysis before accepting the ash.

Fly ash does have a number of beneficial uses. It contains a considerable amount of silica, calcium oxide, and some sodium oxide. This makes fly ash a raw material for cement kilns. It also makes an excellent soil stabilizer for construction projects and barnyards. Because of its potentially hazardous nature, consult with regulatory authorities before using fly ash in such projects.

Handling Limekiln Wastes

The **used refractory bricks** from the limekiln contain chromium, an element on the EPA CERCLA chemical list. Before disposal, companies must have a Toxic Characteristic Leachate Procedure (EPA method 8311) analysis on a sample of the brick. If the leachate contains more than 5 ppm chromium, the waste is a hazardous waste and cannot be sent to an ordinary landfill. Instead, it must be stabilized and sent to a hazardous waste landfill.

Waste limestone fines, generated from screening limestone before calcining, are usable as an aggregate for construction products and roads. Screened coke can be burned in coal-fired dryers and boilers or disposed of in landfills. The discarded CaO and partially burned lime rock can be used for stabilizing soils for constructions projects. The CaO can be recovered by slaking. Lime-slaker wastes can be used as aggregate or disposed of in landfills.

Handling Used-Oil Wastes

Used oil has its own set of government regulations. The two primary ways of dealing with waste oil is to recycle it or burn it for its fuel value. Waste engine oils are laden with heavy metals and should be recycled. Waste gearbox oils do not usually contain hazardous amounts of heavy metals, making them better candidates for burning. Before any decision can be made about burning used oil, the regulatory authorities should be consulted, and all permitting and testing needs to be in place.

When choosing an oil recycler, be certain that the firm is reputable, insured, and licensed. If during the handling, transporting, or recycling operations, the used oil damages property or the environment or harms people, the generator can be held responsible.

One method for reducing waste oil is incorporating an oil-testing program as part of routine preventive or predictive maintenance. At routine intervals, mechanics collect oil samples from gearboxes and engines. The oils are tested for wear metals, breakdown, and contamination. This type of program can reduce the frequency of oil changes to the visible condition of the oil rather than by an hour meter or calendar period.

Another way to minimize wastes is to minimize the amount of materials that become contaminated with oil. Having a secondary containment around oil tanks can prevent contamination from leaks.

Handling Vegetative Wastes

Spoiled or moldy beets, beet pulp, pulp pellets, weeds, and beet chips are **biodegradable wastes**, which can be decomposed with oxygen (aerobically) or without oxygen. These materials do not store long before degrading and should be dealt with soon after they are generated. Temporary storage areas for vegetative wastes should have storm water runoff protection.

Once degraded, these waste materials will provide nutrients and humus to soils. Most of the nutrients in vegetative matter are unavailable for plant growth until microorganisms release

them through a process called mineralization. Because the microorganisms that break down vegetative matter require nutrients, this process can reduce the available nutrients in the soil early in the growing season. As the season progresses, the nutrients become available. Much of the nutrients not available in the first year will become available in subsequent years.

Handling Laboratory and Process Chemical Wastes

Many waste chemicals from both the laboratory and the process are hazardous and must be treated appropriately. Waste pesticides and herbicides must be safely disposed of. Empty containers must be rinsed before disposal. Many wastes that are hazardous due to their reactivity can be easily converted to nonhazardous status by reacting them with appropriate chemicals. Flammables can be burned if permissible and the appropriate equipment is available. If a waste chemical disposal firm is used, be sure it is a reputable firm that follows all the rules.

Hazardous wastes are highly regulated. Any facility generating or storing hazardous wastes must know the rules pertaining to those wastes. Plenty of sources for identifying and dealing with hazardous wastes are available. Chemical manufacturers are a good place to start. Local, state, and federal governments usually have resources you can consult as well. For wastes of uncertain composition, be sure to perform a waste analysis to determine whether the waste is hazardous.

Often, it is safer and less costly to change a procedure, process, or test from one that generates a hazardous waste to one that does not. With modern analytical instrumentation, most laboratory tests can be designed to eliminate or at least reduce hazardous wastes.

Handling Miscellaneous Wastes

Choosing what to do with miscellaneous wastes such as paper, cardboard, scrap metal, and household-type garbage depends on what is locally available, local ordinances, and economics. Recycling is always a good choice if available.

ENVIRONMENTAL MANAGEMENT SYSTEM

To keep track of environmental compliance, modern businesses create an **Environmental Management System** (EMS). Under the EMS umbrella fall not only environmental compliance, but also the environment as part of corporate strategy. This includes a compliance program, environmental audits, and environmental permits.

Managers should develop rapport with the regulators assigned to the facility by the regulatory authority. They should schedule annual meetings to discuss issues of interest to everyone, including local and national environmental issues, capital expenditures, governmental budgets, and the sugar business. Regular meetings should foster respect and professionalism and prevent surprises.

Staying in compliance with all environmental rules requires a concerted effort by everyone at the beet-sugar facility. Every level has responsibilities they must live up to. The board of directors is responsible for providing corporate oversight and letting upper management know that environmental compliance is required. Upper management must make sure the resources are available to maintain compliance. Middle management must design the program and ensure that it meets the company needs. Front-line supervisors must make sure everyone is operating all equipment and following required procedures. Line employees must make sure they operate the equipment and let their supervisors know of any problems with it.

Audits

Every company should conduct regular internal and external environmental audits. The audits need to thoroughly establish that all laws, regulations, and permits are being followed. No matter how good an internal environmental program is, it is a good idea to contract an external agency to conduct an audit every three to five years. Outside auditors make no assumptions and will require you to prove that every aspect of every program is functioning correctly. In addition, they are familiar with compliance programs from a variety of industries and municipalities. They can draw from this experience, making suggestions that can significantly help a program. An additional and important benefit is the independence of the outside auditor.

It is important to quickly remove program weaknesses found by the audit. Managers who do not correct problem areas are negligent. In regulatory or court enforcement actions, problems caused by negligence usually carry higher penalties than accidents.

Permits

Almost everything associated with waste materials including generating, storing, transporting, disposing, and discharging requires permits, permission, and a plan. In addition, if people are using hazardous compounds such as radioactive compounds, sulfur dioxide, or petroleum, they must have permits or create emergency response plans, or do both.

Complying with the regulations drives up costs. The existence of extra regulations increases liability and requires top managers to spend a considerable amount of time working on environmental compliance. If there is a way to legally avoid the regulations, it becomes attractive.

Permitting almost always takes more time than anticipated. Start the permitting process as soon as possible, set a strict timeline, and keep as close to it as possible.

Often, beet-sugar companies hire consultants to help in environmental permitting because their employees lack the required expertise. Consultants can be expensive but can save considerable costs because of their experience.

Because regulators and facility managers have different goals, disagreements can arise. It is always best if these disagreements can be worked out through reasonable discussion and not in the courts.

END-OF-THE-CHAPTER NOTE

In Chapter 10, you learned about air pollutants, liquid wastes, and solid wastes produced by a beet-sugar factory. You also learned guidelines for their optimum treatment. Most importantly, you became familiar with the treatment of wastewaters and their reuse, which reduces potential environmental effects and operational costs of the factory.

CHAPTER 11

SUGAR LABORATORY AND METHODS OF ANALYSIS

SECTIONS

1. Laboratory Organization
2. Laboratory Analytical Instruments
3. Laboratory Reagents
4. Beet-End Methods of Analysis
5. Sugar-End Methods of Analysis
6. Quality-Control Methods of Analysis
7. Special Methods of Analysis
8. Molasses-Desugaring Methods of Analysis
9. Environmental Methods of Analysis
10. Laboratory Safety and First Aid

Note: From this point on, the term **molasses-desugaring process** is used to refer to molasses desugaring by chromatographic process (**MDC process**).

ABOUT THE CHAPTER **Chapter 11** assists sugar chemists in the standardization of their laboratory practices. In general, sugar factories in different countries (even different factories in the same country) manage their laboratories in different ways and have different methods of analysis. The main reason is that these subjects have not been dealt with adequately in the sugar literature. With the development of the sugar industry, the need for a standardization of the organization of sugar laboratories and particularly the methods of analysis used in these laboratories is required. This chapter has accordingly been prepared to cover those requirements.

History: The first **beet-sugar laboratory** was setup by C. Scheibler in Berlin, Germany, in 1867. Since then, the laboratory has been an essential part of every beet-sugar factory.

Beet-Sugar Handbook, by Mosen Asadi

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Figure 11.1 A sugar laboratory from different angles

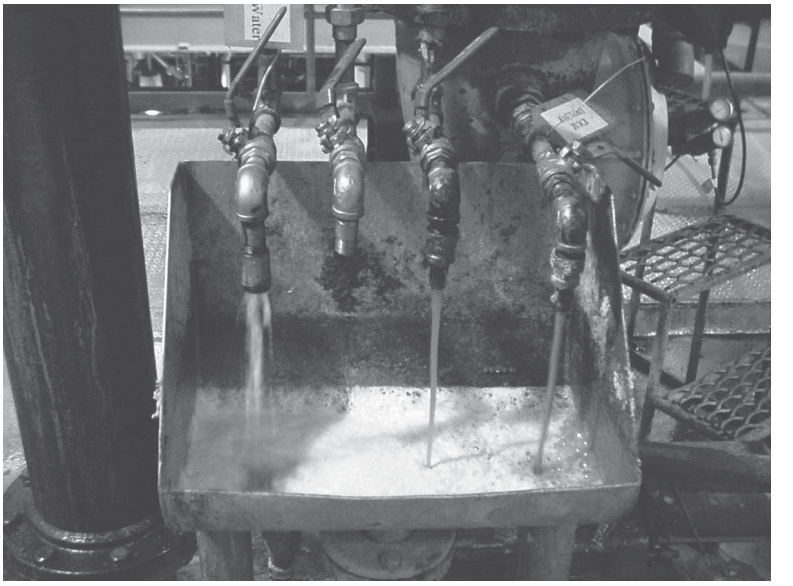


Figure 11.2 Some sample points in a beet-sugar factory

The laboratory (lab) is viewed as the mind of the factory because of its important functions. The main functions of the sugar-factory lab are the following:

- Sample analyses
- Data analyses

To control operations in the sugar factory, the lab analysts collect samples from different steps of the process at different intervals to perform various analyses (see Figure 11.2).

The lab also collects data and every day prepares a **daily report of operation** that helps control factory performance. Figures 11.3 and 11.4 show the daily reports of operation of a beet-sugar factory equipped with two diffusers, thin-juice softening, and molasses-desugaring process (i.e., molasses desugaring by chromatographic process, **MDC process**).

Daily report of operation (beet-slicing operation)

	Day	Week	Todate
Beet sliced (**t**)			
Average slice (**t**)			
Beet purchased (**t**)			
Beet on hand			
% Sugar in cossettes			
Sugar entered (**t**)			
Total sugar produced (**t**)			
Remelt sugar (**t**)			
Net sugar produced (**t**)			
Net extraction, % on sugar			
t Sugar / t Beet			
Sugar:			
Color			
Screen			
Moisture			
Losses:			
Sugar to molasses (**t**)			
" " , (**% OB**)			
Sugar to pulp (**t**)			
" " , (**% OB**)			
Sugar to limecake (**t**)			
" " , (**% OB**)			
Accounted losses (**t**)			
Accounted losses (**% OB**)			
Unaccounted losses (**t**)			
Unaccounted losses (**% OB**)			
Molasses and pulp:			
Molasses produced (**t**)			
" " , (**% OB**)			
Pulp-pellets produced (**t**)			
" " , (% OB)			
Limekiln:			
Limestone used (**t**)			
" " , (**% OB**)			
Coke used (**t**)			
" " , **%** on Stone			
Energy used:			
Boilers gas (m^3)			
" " , (**% OB**)			
Dryers gas (m^3)			
" " , (**% OB**)			
Dryers gas (m^3/**t pellet**)			
Total gas			
" " , (**% OB**)			
Steam (**% OB**)			
kg Steam Produced / m^3			
Electric power used (**KWH**)			
KWH / t Beet			

Continued

Draft
Knife used
Diffusion juice, RDS
Diffusion juice, purity
Thin juice, RDS
Thin juice, purity
Thick juice, RDS
Thin juice, color
Thick juice, color
Fresh water, pH
Press water, pH
Diffusion juice, pH
Mixed juice, pH
Pre-carb juice, pH
Inter-med Juice, pH
First carb, pH
Second carb, pH
Thin juice, pH
Thick juice, pH
Purification:
Alkalinity, first carb
Alkalinity, second carb
Alkalinity, pre-carb
Alkalinity, int-med
Limesalts, hard juice
Limesalts, hard Jc.(S. ashed)
Limesalts, soft juice
Limesalts, molasses
Milk of lime, Baume
Mud to mixed Jc., Baume
Settling rate
Mixed juice, %CaO
Pre-carb, %CaO
First carb, %CaO
% Lime to inter-med. liming
% Lime to main liming
Limecake, %sugar
Limecake, %CaO
Limecake, % moisture
Tons CaO produced
% CaO on Beet
CaO / NS in diffusion juice
% NS elimination
Pulp & Pellets:
Wet pulp # 1, %sugar
Wet pulp # 2, %sugar
Press pulp, % sugar
Press water, %sugar
Pellets, %sugar
Wet pulp, %sugar
Pressed pulp, %moisture
Dry pulp, %moisture
Pellets, %moisture

Continued

Standard liquor:
RDS
Purity
pH
Color
White pans:
Number of pans
Pans, m^3
Mass RDS
Mass purity
Mass, pH
kg / 100 m^3
Boiling drop
Hi-green, RDS
Hi-green, purity
Hi-wash, RDS
Hi-wash, color
High-raw pan:
Mass RDS (chamber #4)
Mass purity
Mass, pH
% Crystal in mass
Boiling drop
Low green, RDS
Low green, purity
Hi-raw sugar, Pol
Hi-raw sugar, Color
Hi-raw seed magma, RDS
Hi-raw seed magma, purity
Low-raw pan:
Mass RDS (chamber #4)
Mass purity
Mass pH
Low-raw sugar Pol
% Crystal in mass
Boiling drop
Extract to sugar-end, RDS
" " purity
Molasses:
RDS
Purity
pH
Color
Temperatures:
% Evaporator efficiency
Diffusion water, temperature
Tower #1, temp.
Tower #2, temp.
Diffusion juice temp.
First carb temp.
Second carb temp.
Standard liquor temp.

Figure 11.3 Daily report (beet-slicing operation)

Daily report of operation (MDC-process operation)

	Day	Week	Todate
Day of operations			
Molasses processed (**t**)			
Molasses inventory (**t**)			
Granulated sugar produced			
Sugar to sugar-end from storage tank			
Sugar to sugar-end from desugarization			
Total sugar to sugar-end			
Gross sugar produced (**t**)			
Remelt sugar (**t**)			
Net sugar produced (**t**)			
Sugar-end extraction (%)			
Granulated sugar, color			
" " , screen(% on 35)			
" " , moisture			
Predicted production			
Gran. sugar (**t**), @ purity=60, RDS=80			
t sugar/t molasses desugared			
Extract molasses (**t**)			
Extract			
Produced (**t**)			
To sugar-end from storage tank			
Total to sugar-end (**t**)			
Inventory (**t**)			
Betaine			
Produced (**t**)			
Shipped (**t**)			
Inventory (**t**)			
Betaine recovery, %			
CMS			
Produced (**t**)			
Shipped (**t**)			
Inventory (**t**)			
Extract molasses			
Produced, tons			
Shipped, tons			
Inventory, tons			
Sugar balance			
Molasses (**t**)			
Extract (**t**)			
Betaine (**t**)			
CMS (**t**)			
Unaccounted (**t**)			
Unaccounted, %/sugar entered			
Separator recovery (**%**)			
Solids balance			
Molasses (**t**)			
Extract (**t**)			
Betaine (**t**)			
CMS (**t**)			
Unaccounted (**t**)			
Unaccounted, %/solids entered			
Energy			
Gas to boilers (m^3)			
m^3 / ton molasses			
Steam, % on molasses			

Continued

	Day	Week	Todate
Molasses, RDS			
" Purity			
" Color			
Diluted Molasses, RDS			
" " pH			
" " L. Salts, meq/DS			
" " Sediment, ppm			
Upgrade, RDS			
" Purity			
" Color			
" pH			
Conc. Upgrade, RDS			
" " Purity			
" Color			
" pH			
" Sediment			
Betaine, RDS			
" , Purity			
Concen. Betaine, RDS			
" " Purity			
" " pH			
Raffinate, RDS			
" , Purity			
Conc. Raffinate (CMS), RDS			
" " , Purity			
" " , pH			
Extract, RDS			
" Purity			
" Color			
" , pH			
Conc. Extract, RDS			
" " , Purity			
" " , Color			
" " , pH			
Extract, Temperature			
Special tests			
Molasses, betaine (%/DS)			
" invert (%/DS)			
" GC sucrose			
" true purity			
Extract, invert (%/DS)			
" GC sucrose			
Stored extract, pH			
" " invert (%/DS)			
Betaine fraction, betaine Pur. (%/DS)			
" " invert (%/DS)			
" " GC sucrose			
CMS, betaine (%/DS)			
" GC sucrose			
" " GC sucrose			

Figures 11.4 Daily report (MDC operation)

The material balance of the factory is reported on a weekly basis and is called the **weekly report of operation**. Unlike the daily report, the weekly report considers the **carry-in stock of sugar** (the mass of equivalent sugar in individual tanks) in process. Stocktaking determines the quantities of equal sugar and molasses, which would be produced from the stock. The lab also prepares the **end-of-campaign report**, which is the final report of operations for one campaign.

The lab operates three eight-hour shifts (3×8 hours), seven days a week, during the **beet campaign** (sugarbeet operational season). The campaign usually lasts between three and seven months, depending on the quantity of sugarbeets available for processing.

The **quality-control** section of the lab is active year-round, and tests the quality of the **final products** (granulated-refined sugar, powdered sugar, brown sugar, and other specialty sugars). The number of shifts in this section depends on the station's schedule for production of the specialty sugars.

The **environmental** section of the lab is also active year-round on an eight-hour day basis. The environmental analyst analyzes wastewater samples from the factory's wastewater-treatment ponds and other related sources.

CHAPTER
11

SECTION 1

LABORATORY ORGANIZATION

Beet-sugar laboratories usually have different organizational and management structures. The organization described in this section has efficiently served one U.S. beet-sugar factory for many years. In this type of organization, the analyses are divided in the following five groups:

- Beet-end analyses
- Sugar-end analyses
- Quality control analyses
- Special analyses
- Molasses-desugaring analyses (for factory equipped with the MDC process)
- Environmental analyses

The title of each **lab analyst** (bench chemist) corresponds to the type of analyses that the analyst performs. For example, the **beet-end analyst** is responsible for testing all samples taken from the beet-end section, starting with analyses on the cossettes all the way to tests on the thick juice.

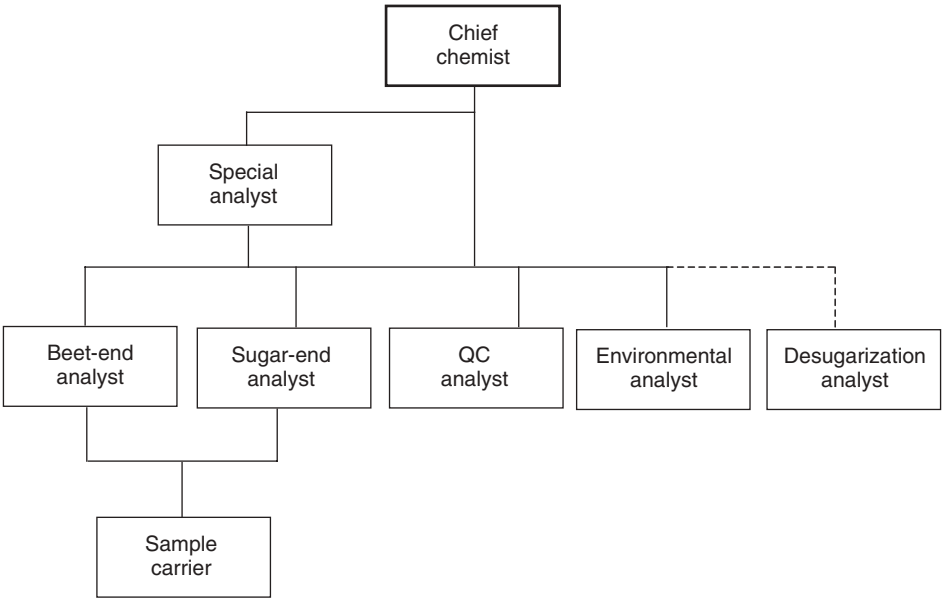


Figure 11.5 Lab organization

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The **sugar lab** is managed by a **chief chemist**, whose job description includes the following:

- Managing the lab and supervising lab employees
- Supervising analysts on the lab's analytical procedures
- Coordinating lab work with factory personnel to avoid disrupting factory efficiency
- Presenting process accounting and operational reports
- Resolving with the factory environmental manager wastewater issues

In the past, the chief chemist also took care of all the factory's environmental issues. However, today's environmental workloads have required the creation of an environmental manager position. Therefore, most sugar factories employ an environmental manager to supervise wastewater treatment practices, and to communicate and deal with environmental agencies such as the U.S. Environmental Protection Agency (EPA) in matters regarding wastewater reports, wastewater discharge permits, and other environmental issues.

The environmental section of the lab is responsible for performing analytical tests under the chief chemist's supervision. The test results are then reported to the environmental manager for further considerations.

The chief chemist usually has an assistant. In some labs, the **special analyst** functions as the chief chemist's assistant. The special analyst's job description includes the following tasks:

- **Collecting data:** These data include cossettes scale readings (for measuring the amount of beets processed), amounts of molasses and pulp production, and levels of usage of fuel, limestone, coke, and other related data.
- **Preparing daily report of operation:** Most sugar factories use an in-house software to prepare the daily report of operation (refer to the Appendix for more information). The daily report's preparation takes about half a day of the special chemist's time, leaving half a day free to analyze special samples. Special samples are analyzed on an as-needed basis only, so a test can be scheduled on any day of the week. Otherwise, one day may be allocated for analyses of true sucrose by the inversion method on a weekly composite sample of molasses, while another day may be allocated for microbiological tests on the weekly composite sample of sugar.

Note: Usually, an assisting analyst is trained to perform the duty of the special analyst on his or her days off. The analyst titles and the personnel needed (in parenthesis) are as follows:

- Special analyst (one per day)
- Sample carrier (one per shift)
- Beet-end analyst (one per shift)
- Sugar-end analyst (one per shift)
- Quality control analyst (one per shift)
- Environmental analyst (one per day)
- Molasses-desugaring analyst (one per shift, when the factory has desugaring process)

Besides the three eight-hour shifts, the lab employs additional analysts for each position, who work on a rotational basis, known as the **swing shift**, to cover days off for the other analysts.

The **beet-end analyst** is responsible for running all samples that come from the **beet-end section** (the processing section up through juice evaporation), starting with analyses on cossettes all the way to the tests on the thick juice (the product of the evaporation station). In the same way, the **sugar-end analyst** runs all sugar-end samples, such as analyses on standard liquor, on massecuites from the first, second, and third stages of crystallization, and on molasses.

CHAPTER
11

SECTION 2

LABORATORY ANALYTICAL INSTRUMENTS

SUBSECTIONS

- Refractometer
- Polarimeter
- pH Meter
- Spectrophotometer
- Conductometer

ABOUT THIS SECTION **This section** explains the most important analytical instruments used in sugar labs: refractometer, polarimeter, pH meter, spectrophotometer, and conductometer (conductivity meter). Notes, at the end of each subsection, explain practical issues that chemists might encounter while using these instruments in routine analytical tests.

REFRACTOMETER

HISTORY

In 1768, A. Baume, a French chemist, invented a hydrometer for measuring the amount of dry substance (*DS*) in a solution by mass percentages. The 0-mark on the scale indicated the point at which the hydrometer sinks in water, and a 15-mark the point at which it sinks in a 15% salt solution.

In the 1840s, K. Balling (1805–1868), a Czech chemist from Prague, invented the hydrometer scale (degree) to measure the *DS* of sugar solutions. The scale then was named the Balling scale. The Balling hydrometer indicated the concentration of sugar solutions by mass percentage. Balling also formulated the purity equation, which has greatly served the sugar industry.

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In 1854, A. F. W. Brix, a German chemist, revised the Balling scale. The new scale became known to sugar technologists as degree Brix (or simply Brix). A hydrometer scaled in such way was called a Brix hydrometer, which is still used in the sugar industry.

In late 1800s, E. Abbe invented the refractometer. The dry substance measured by the refractometer is known as % refractometric dry substance (% *RDS*), which has the same meaning as Brix. In this book, the term *RDS* or simply *DS* is used.

The refractometer measures the dry-substance (*DS*) content in a solution sample. The technique used by the refractometer is called **refractometry**, which is based on the angle of refraction (bending) of a beam (ray) of light when it strikes a different medium (see Figure 11.7). As the beam passes from air through a solution, its direction changes, creating an angle called the **refraction angle** or the **refractive index** (RI, the refractive index of pure sucrose solutions is given in Table A.14 of the Appendix). Because of the direct relation between RI and *DS*, the refractometer displays the results in *DS*, referred to as *RDS* (refractometric dry substance) or traditionally **Brix**. Refractometers are calibrated in a way to indicate the dry substance *(DS)* of the sample in mass percentage (% m/m).

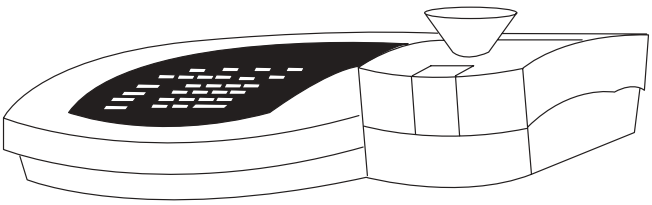


Figure 11.6 A modern refractometer

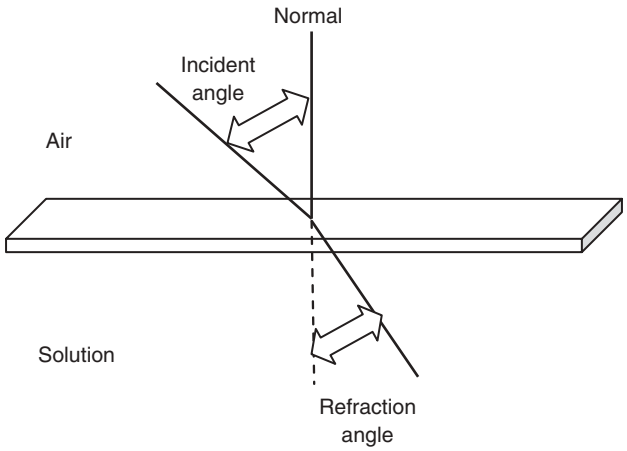


Figure 11.7 The basics of refractometry

The RI of a solution is the ratio of the speed of light in air to the speed of light in the solution. The RI of air is equal to 1 (no refraction takes place), the RI of pure water at 20°C and a wavelength of 589 nanometers (nm = 10^{-9}m) is 1.333, and the RI of a 10% pure sucrose solution is 1.348. Therefore, the RI relates directly to the concentration of the *DS* in the sample. The following affect the RI:

- Temperature of the sample
- Concentration of the sample
- Wavelength of the refractometer

The dry substance (*DS*) measured by the refractometer is called a **refractometric dry substance** (*RDS*), which has the same meaning as Brix**.** *RDS* represents the percentage of *DS* of a solution by mass (g per 100 g of solution) measured by refractometer. Note: Sugar scientists recommend the use of term *RDS* or simply *DS* instead of Brix (see the Sugar Terminology section of Chapter 1).

The *RDS* of a pure sucrose solution represents the **exact dry substance**. But this is not so if the solution is an impure sucrose solution due to the presence of nonsucrose (nonsugar) substances (all soluble substances except sucrose), which affect the RI and consequently the *RDS* differently. For example, **invert sugar** (the 50 : 50 solution of glucose and fructose solution) decreases the RI, and raffinose and dextran increase the RI. In general, the different nonsugars in the **in-process products** (juices, syrups, and liquors) can be said to have opposite effects on the IR (one may increase it, while the other decreases it), to the point where they cancel each other out. However, the *RDS* of an impure sucrose solution represents the **apparent** (approximate) **dry substance** (practical dry substance) of that solution. The **true dry substance** is measured by the **oven-drying method**.

In sugar laboratories the refractometric method is used for almost all samples for the following two good reasons:

- The results measured by the refractometric method and by the drying method are very close.
- Determination is faster by the refractometric method (about 3 minutes) compared to the oven-drying method (about 3 hours).

In addition, in-process products and molasses resist drying. Therefore, the refractometric method is used in all practical purposes in today's sugar-processing operations.

Like a refractometer, the **hydrometer** works based on the density of the solution sample to measure the percentage of dry substance in sugar solutions by mass percentage (% m/m). Hydrometers are made of glass, with a weighted float at the bottom. When a hydrometer is placed in a cylinder containing water, it sinks to the 0 scale; when it is placed in a sample, the depth to which it sinks indicates the *DS* concentration of the sample. Hydrometers are usually calibrated at 20°C, so a correction must be applied when used at any other temperature.

Brix and Baume hydrometers (named after their inventors) are the most-used hydrometers in sugar industry. Baume hydrometers are graduated in Baume scale or simply Baume (Be), at a specified temperature and are mainly used in beet-sugar factories to measure the *DS* content of milk of lime (MOL). (Measurement of the *DS* content of a sample by refractometer is more accurate and faster than by the hydrometer.)

NOTES ON REFRACTOMETRY

- **Refractometers** are calibrated and verified by using a pure sucrose solution at the standard lab temperature (20°C). Therefore, the accuracy of the measurement depends on the temperature of the sample under the test. Warm samples should be cooled to the standard temperature in a water bath before measuring the *RDS*. If the measurement is carried out at a temperature other than the standard, the *RDS* reading must be adjusted accordingly. The following values are used for adjustment:

 For each degree above 20°C, add 0.08 to the *RDS* reading

 For each degree below 20°C, subtract 0.08 from the *RDS* reading
- When measuring *RDS*, allow about 30 seconds for the sample placed on the prism of the refractometer to reach the temperature of the prism.
- Automatic temperature compensation (ATP) is a device for temperature correction. Newer refractometers are equipped with this device, which detects the temperature between the sample and the sample compartment of the refractometer. Remember that refractometers with ATP are still equipped with a water-bath circulator port to operate them under standard temperature.
- New refractometers are easy to use. You simply place a few drops of the sample on the prism (covering the prism with a thin layer of sample) and push a button to see the % *RDS* on a digital display.
- Unlike the old refractometers, in which operators had to perform line-matching, newer ones minimize the possibility of human error.
- To **calibrate** a refractometer, use deionized water (**DI water**, soft water prepared by passing city water through ion-exchange resin) to set 0 and a known sugar solution (e.g., a 20% by mass solution) for a second point of calibration. The calibration should be performed at standard temperature (20°C).
- You can also use a liquid reference standard (calibration liquid) for **calibration**. This liquid can be purchased from laboratory chemical suppliers such as Fisher Scientific and VWR Scientific.
- High concentrated samples (above 60% *RDS*) such as molasses should be double-diluted for better accuracy. Such a sample is also called a **fifty-fifty solution** (50:50 solution), which is usually made by mixing 50.0g sample with 50.0g of deionized (DI) water. Then, the refractometric reading resulted from 50:50 solution is multiplied by 2 to find the actual *RDS*.
- Because suspended solids in the sample affect the RI, the samples containing suspended solids (such as diffusion juice) should be filtered through a coarse filter (e.g., piece of centrifugal screen) before measuring the *RDS*.
- The refractometer prism should be cleaned with DI water after each test and with soapy water at the end of each shift. While cleaning, care must be taken to avoid scratching the prism surface.
- Spare light bulbs should be available in case a refractometer needs a new light source. When changing the bulb, you must hold it with a soft tissue because fingerprints can change its intensity.
- Abbe, Bausch and Lomb, Belingham + Stanley (BS), Index Instrument, Leica, Rodulph Analytical, Reichert, Schmidt & Haensch, Spectronic, and Zeiss are some of the refractometer manufacturers in today's market.

POLARIMETER

HISTORY

In 1840, Biot, a French physicist, built the first **polarimeter** using polarized light. The polarimeter (Figure 11.8) is one of the most important instruments in the sugar labs.

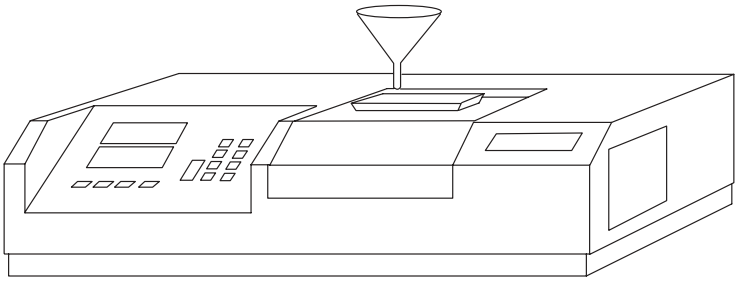


Figure 11.8 A modern polarimeter

The **polarimeter** (polaroscope) measures the optically-active substances (e.g., sucrose) in a solution sample. The technique used by the polarimeter is called **polarimetry**, which is based on the **rotation** (bending) of **polarized light** (the light after passing a polarizer) as it passes through an optically active solution (e.g., a sucrose solution). Light as we usually see it consists of **waves** (amplitude [maximum disturbance during one wave] that changes cyclically) spread in all directions (planes). As light passes through a polarizer, a **polarized light** (a light with a narrow band width in one direction) is formed. If a solution sample changes the angle of polarized light, this indicates the presence of an optically active substance in the sample. The degree of rotation of the polarized light is called the **degree of polarization** (simply **pol**). The degree of polarization (pol) is proportional to the concentration of the optically active substances (such as sucrose) in a solution sample. This property is used in polarimetry.

The following factors affect the degree of polarization:

- Temperature of the sample
- Wavelength of the polarimeter
- Length of the polarimeter tube (tube's path length)
- Nature of the nonsucrose substances in the sample
- Concentration of optically active substances in the sample

The degree of polarization increases with increasing temperature. In sugar labs, the temperature, the wavelength, and the length of polarimeter are kept constant, and clarifying reagents are used to precipitate nonsucrose substances. Therefore, the degree of polarization relates directly to the concentration of sucrose in the sample.

A simple polarimeter (old type) consists of the following, as shown in Figures 11.9 and 11.10:

- A polarizer to create the polarized light
- A tube to hold the sample in the way of the polarized light
- A second polarizer (called analyzer), installed after the sample tube, to create the degree of polarization
- A scale (calibrated 0 to 360°) for measuring the degrees of polarization.

Unlike older models, modern polarimeters convert the degrees of polarization to numerical values and display directly **% polarimetric sucrose** (*PS*). Polarimetric sucrose sugar expresses the sucrose content by mass, which has the same meaning as **degree of polarization** (simply

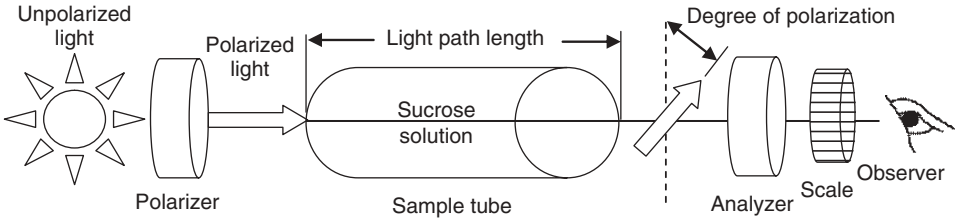


Figure 11.9 Basic parts of a polarimeter

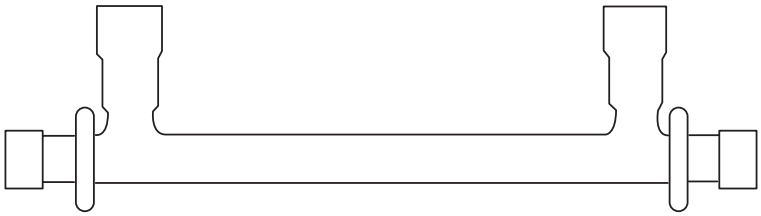


Figure 11.10 Polarimeter sample tube

pol), which is the degree of rotation of the polarized light in a sugar solution. Remember that in this book, **% polarimetric sucrose** (simply % sucrose content) is used, not pol (see the Sugar Terminology section of Chapter 1).

In polarimetry, some substances rotate the polarized light to the right, and some rotates the light to the left. The direction of rotation is indicated by a plus (+) for a right-rotation, and a minus (–) for a left-rotation. For example, sucrose, glucose (dextrose), raffinose, dextran, starch, and invert sugar rotate the polarized light to the right and so are called **right-hand sugars** (dextrorotatory). Fructose (levulose) rotates polarized light to the left and therefore are called **left-hand sugars** (levorotatory). Following is the specific rotation of a normal solution (explained later) of some sugars at 20°C and a wavelength of 589 **nanometers** (nm, 1 m = 10^9 nm):

Sucrose	+66.5°
Glucose	+52.5°
Fructose	–92.4°
Raffinose	+123.2°

In sugar labs, the sucrose content is measured by two methods:

- Inversion method
- Polarimetric method

However, the polarimetric method is usually used because

- The results measured by the polarimetric method and by the inversion method are very close.
- Determination is fast by the polarimetric method (about 3 minutes), and slow by the inversion method (about 3 hours).

Testing impure sucrose solutions by the polarimetric method requires applying a clarifying reagent to the sample before it is tested by the polarimeter. The reasons are given next.

Clarifying Reagents

Clarifying reagents are chemicals used in polarimetry for the following purposes:

- To precipitate active substances, which interfere with polarimetric tests
- To produce a clear sample, which is a prerequisite for polarimetric tests

In-process products (juices, syrups and liquors) of the sugar factory are impure sucrose solutions. The polarimetric results obtained from these solutions are affected by the amount of **impurities** (nonsucrose substances), since many substances (other than sucrose) are also optically active and change the direction of polarized light. These substances are considered sources of error in the polarimetric measurements. To reduce the effects of these interfering substances, sugar labs use **clarifying reagents** (e.g., aluminum chloride) on the samples to precipitate nonsugars. Then the precipitate is filtered, and the percentage of sucrose in the **filtrate** (the liquid collected during filtration) is measured by polarimeter.

The effectiveness of clarifying reagents in eliminating active substances is not complete, however; for example, aluminum salts do not completely precipitate raffinose. As a result, sucrose measurement by polarimeter does not exactly reflect the sucrose content if the sample is an impure sucrose solution. In impure solutions, the polarimetric sucrose (*PS*) represents the **approximate sucrose content**. The **exact sucrose** is measured by the **inversion method**. Determination of *PS* is fast (about 5 minutes) by the polarimetric method but time-consuming (about 5 hours) by the inversion method. Nevertheless, the polarimetric method is accurate enough to be used in almost all practical purposes in today's sugar-lab tests. To distinguish the test methods, the result of polarimetric sucrose is called **direct pol**. Based on these facts, two terms are used in the sugar industry to express sucrose content:

- **Approximate sucrose content:** It is measured by the polarimetric method and is called polarimetric sucrose (*PS*)
- **Exact sucrose content:** It is measured by the inversion method and is called true sucrose (*TS*)

The sugar industry has used various chemicals as clarifying reagents. **Lead acetate** was used for many years in every sugar lab. It worked well, but its **health** and **environmental** factors led many countries to prohibit its use in last two decades. Therefore, sugar labs have been using **nonhazardous chemicals** as clarifying reagents, such as a combination of aluminum chloride and ammonium hydroxide or aluminum sulfate combined with ammonium hydroxide. These chemicals are not considered toxic and do not require a special environmental disposal process.

The quality and quantity of clarifying reagents should be chosen to create the following results:

- A sample with high clarity and a high filtration rate
- Consistent results identical to those using lead acetate

An **anion–ion exchange resin** combined with **activated carbon** was also used as a clarifying reagent, but it is no longer popular because of complications regarding the moisture content of

the resin and the requirement for fine filtration to filter activated carbon. In addition, activated carbon needs special solvents for cleaning, which causes safety issues.

Some labs use brand reagents supplied by sugar chemical suppliers. One of these reagents is the Octopol (ABC) sugar clarifier, which is a mixture of three fine powdered chemicals. These chemicals should be mixed together thoroughly at least three days prior to use to be activated. Otherwise, the sample becomes thicken and difficult to filter. This reagent generates good clarity and consistent results with a high correlation compared to lead acetate, but the thorough preparation and usage should be followed.

A compound, combined from aluminum chloride hexahydrate ($ALCl_3.6H_2O$) and ammonium hydroxide (NH_4OH), is used as the clarifying reagent in the analytical procedures in this book for the following reasons:

- Hydrated aluminum chloride dissolves quickly and furnishes an aluminum ion, which is necessary for further reactions with nonsucrose substances
- Sugar measurements obtained from this reagent have a high correlation compared to lead

Polarimeter Calibration

The International Commission for Uniform Methods of Sugar Analysis (**ICUMSA**) recommends the following for polarimeter calibration:

- A **sucrose normal solution** (26.00 g of pure sucrose diluted in distilled water to a final volume of 100 ml) should be used for adjusting the 100-scale of the polarimeter
- A **standard temperature** (20°C) should be maintained during the test
- The **wavelength** of the polarimeter must be set at 589 nm

Under these conditions, the optical rotation of a normal sucrose solution (26.00 g of the sample diluted to 100 ml at 20°C) polarized in a 200-mm polarimeter tube at 20°C is 66.54, the same as the 100-scale of the polarimeter. The scale of the polarimeter (0 to 100) determined under this condition is referred to as % polarimetric sucrose (*PS*).

Although the polarimetric samples are prepared in % mass per volume (% m/v), the results are in % by mass (% m/m). The reason is that the instrument compares the degree of rotation of the sample with data in its memory for normal mass. In other words, the polarimeter compares the degrees of rotation based on two masses.

As stated, polarimeters are calibrated based on the rotation of the **normal sample** (26.00 g of sample diluted to 100 ml at 20°C) and using a 200-mm polarimeter tube. In this case, the percentage of polarimetric sucrose (simply % sugar) is

$$\%\ \text{Sugar} = \text{Polarimetric reading} \tag{11.1}$$

When using different sample weight or different-size polarimeter tube, you should equate the polarimetric reading to the polarization at normal weight by using the following equation:

$$\%\ \text{Sugar} = \text{Polarimetric reading} \times \frac{26.0}{\text{Mass}_{\text{Sample}}} \times \frac{200}{\text{Tube}_{\text{Length}}} \times \frac{\text{Flask}_{\text{Volume}}}{100} \tag{11.2}$$

For example, if **half-normal sample** (13.0 g to 200-ml flask) and a 100-mm tube is used, the polarimetric reading is multiplied by 8 to find the percentage of sucrose:

$$\% \text{ Sugar} = \text{Polarimetric reading} \times \frac{26.0}{13.0} \times \frac{200}{100} \times \frac{200}{100} = \text{Reading} \times 8$$

You should also be aware of the following factors:

- Sample with no dilution (the sample is polarized directly without dilution) and using a 200-mm tube:

$$\% \text{ Sugar} = \text{Polarimetric reading} \times 0.26 \quad (11.3)$$

- Sample with no dilution and using a 100-mm tube:

$$\% \text{ Sugar} = \text{Polarimetric reading} \times 0.52 \quad (11.4)$$

NOTES ON POLARIMETRY

- **Polarimeters** are **calibrated** and verified using a normal solution of sucrose (as explained earlier) or a **quartz control tubes**. The quartz tubes are more reliable for calibration than the normal solution of sucrose because the values of the tubes are not subject to change. Every sugar lab should have two quartz control tubes for polarimeter verification. One tube has a sugar value of 100% and the other has the same value order as the most samples (around 20). Quartz tubes can be purchased from polarimeter manufacturers.
- The **weight** of dark samples (e.g., molasses and low-green syrup) may be necessary to reduce to half normal (13.0 g to 200 ml) and read the sample using a 100-mm tube. Then, the polarimetric reading is multiplied by 8 to find the percentage of sugar content.
- **Samples** should not be diluted beyond half normal unless the sample is extremely dark, since then the multiplying factor in the polarimeter reading becomes high and magnifies any error.
- **Air bubbles** are released from the tube by tilting it to one side while filling.
- A **sugar flask** (Kohlrausch flask) is recommended for diluting the samples because its wide top makes it easier to discharge the sample from the flask.
- **Samples containing particulate materials** (e.g., diffusion juice) should be filtered through a coarse filter (e.g., a piece of centrifuge screen) before being weighted for the polarimetric test.
- A **standard temperature** (20°C) is required for polarization. Samples should be cooled down in the water bath to this temperature before polarimetric reading because optical rotation increases with a rise in temperature, causing of a faulty result.
- A **temperature sensor** is a build-in device in some newer polarimeters. The sensor measures the sample temperature to make corrections to the standard temperature (normally 20°C and 27.5°C for tropical areas). In any case, cooling the samples to standard temperatures before the polarimetric reading is recommended because the sensor might not be correctly calibrated for temperature.
- A **preventive service** by the polarimeter manufacturer (at least every two years) is recommended to maintain the polarimeter's integrity. This service includes general maintenance and calibration of the polarimeter and verification of the quartz control tubes.
- **Polarimeter tube length** refers to **path length** (light path), which is the distance between the cover glasses on each side of the polarimeter tube. The path length is important in polarimetry because it is proportional to polarization.
- A **100-mm polarimeter tube** is used for sugar determination and the calculation of all analytical methods in this book.

- The **polarimeter tube** should be rinsed twice with the sample and then filled with the sample for polarimetric reading. It should be washed well with distilled water after each test.
- The **polarimeter tube** should be disassembled and cleaned promptly with detergent or 5% acetic acid followed by a rinsing with distilled water at the end of each shift. If a flow-through tube is used, it should be flushed with the same solutions.
- The **polarimeter tube** consists of a tube body, a glass-cover for each end, a rubber washer for each end, and a metal end-cap for each end. The wrong assembly changes the path length, which is critical to the polarimetry. Remember that the polarimeter tube should be assembled in the following order:
 - Tube body
 - Glass cover
 - Rubber washer
 - Metal end-cap
- The **metal end-cap** should be screwed loosely (a finger-tight pressure is sufficient).
- The **glass cover** of the polarimeter tubes should be checked for scratches and replaced if damaged. Damaged glass can result in errors.
- The **flow-through tube** (flow cell) minimizes sample handling and reduces measurement time. The tube is connected from one side to the polarimeter tube mounted in the sample compartment and from other side to a waste container. The analyst simply pours the sample into the funnel, reads the polarimetric result, and flushes the funnel with either water or the next sample.
- **Glass windows** on each side of the sample compartment of the polarimeter should be cleaned once a week. The glass cover must be cleaned with soapy water and dried with a soft cloth.
- **Beet juice** always contains more **glucose** than **fructose**. Therefore, if these substances are not clarified completely by using suitable clarifying reagents, the sample indicates a higher value because sucrose and glucose rotate polarized light to the same direction. (Sucrose and glucose are right-hand sugars and fructose is a left-hand sugar.)
- **Glucose** behaves in a strange way with regard to its optical rotation during polarimetry. The specific rotation of a freshly prepared glucose solution slowly changes until it stabilizes with time. This phenomenon is known as **mutarotation** (existing of two isomers of a sugar in a solution, so it takes some time for these isomers to become in equilibrium situation). Therefore, it is recommended that the sample be allowed to set for about three minutes before a polarimetric reading.
- **Invert solutions** (solutions in which the sucrose is inverted to an equal quantity of glucose and fructose by acid) should be made up and polarized at exactly 20°C because their specific rotation varies greatly with the temperature.
- **Invert solutions** are subject to a rapid change; therefore, the polarimetric reading should be taken when the polarimeter display is stabilized. It is recommended that inverted samples be allowed to set for 3 minutes before a polarimetric reading.
- **Raffinose** has almost twice (exactly 1.8 times higher) the specific rotation of sucrose (both raffinose and sucrose are in the right-hand group). As a result, the polarization of granulated-refined sugar containing raffinose might exceed the polarimetric reading above the 100° scale.
- **Raffinose** present in sugarbeet increases the polarimetric reading of the cossettes test. This causes a wrong report in sugar entered and, consequently, the material balance of the factory. Some sugar factories discredit any sugar that entered the factory when the raffinose content of the beets is extremely high.
- **Dextran** has about three times the specific rotation of sucrose; therefore, its presence in the sample results in a false polarimetric reading.
- **Normal sucrose solution** has a **specific rotation** of +66.54 at 20°C and at a wavelength of 589 nm. This expression is still used by some customers as a specification for the quality evaluation of

granulated-refined sugar, since old polarimeters read the angle of rotation and were not be able to translate it to the sugar value. Modern polarimeters do not indicate rotation values because the rotation is directly translated to the percentage of sucrose content. If you need the specific rotation, an equation ratio between the sugar value of the pure normal sucrose solution (66.54°) and the sugar value of the sample can solve the problem. For example, if the polarimeter reading of a normal sample of sugar was read 99.94, the specific rotation of the sugar sample is

$$66.54 \times \frac{99.94}{100} = 66.5$$

- The **measurement at two wavelengths** (589 nm and 880 nm) is a valuable feature of some modern polarimeters. The wavelength of 589 is for ordinary samples treated with clarifying reagents. The wavelength of 880 (near infrared, **NIR**) is above the absorption level of most dark-sugar factory samples, allowing the measurement of less-clarified or nonclarified, but filtered, samples.
- A **purity program** is software offered by some modern polarimeter manufacturers for communication between the polarimeter and the refractometer for purity calculations. This program can calculate the purity by mass or mass per volume (m/v) because the polarimeter may have one type of dilution and the refractometer another.
- **Statistics**, such as the average and standard deviation of measurement values, can be calculated by some modern polarimeters. This feature is particularly helpful for research.
- **Extra bulbs** should always be available in case the polarimeter needs a new light source. When changing the bulb, you must hold it with a soft tissue because fingerprints can change the optical response.
- The **air filter** of the polarimeter should be washed once a week in warm water containing a detergent.
- Abbe, Bausch and Lomb, Belingham & Stanley (BS), Index Instrument, Leica, Rodulph Analytical, Schmidt & Haensch, Spectronic, and Zeiss are some of the polarimeter manufacturers in today's market.

pH METER

The **pH meter** measures the pH in a solution sample. The technique used by the pH meter is called **potentiometry**, which is based on the potential difference between a pair of electrodes placed into a solution from which the **activity of an ion** (pH) can be determined. The activity of ions in a solution is important because it determines the rate of reactions. Simply speaking, pH is a term for expressing the acidity or alkalinity of a solution. The pH value is a dimensionless number (data with no unit) expressed on a scale of 1 to 14, with 7 the neutral value (see Figure 11.11). Acids have a pH from 1 to 7 (*the higher the pH, the weaker is the acid*). Bases (alkalis) have a pH from 7 to 14 (*the higher the pH, the stronger is the base*).

Some acids such as **acetic** ($HC_2H_3O_2$) and **boric** (H_3BO_3) are weak; other acids such as **sulfuric acid** (H_2SO_4) and **nitric acid** (HNO_3) are strong. Likewise, bases can be weak, such as **ammonia** (NH_3), or strong, such as **caustic** (sodium hydroxide, NaOH).

Chemically speaking, pH is defined as the negative to the base 10 of the **hydrogen-ion concentration** (H^+) of a solution (see Section 2 of Chapter 12 for review of "log)":

$$pH = -\log(H^+) \tag{11.5}$$

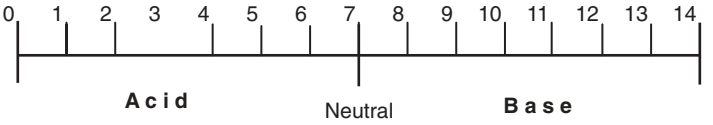


Figure 11.11 pH scales

Each integer difference represents a 10-fold increase in hydrogen ions as pH gets smaller and a 10-fold decrease in hydrogen ions as pH gets larger.

EXAMPLE 1

Given:

Molality (M) of H^+ 10^{-7} mol/L

Calculate:

pH

$$pH = -\log(H^+) = -\log 10^{-7} = 7$$

In any aqueous solution at 25°C, no matter what it contains, (H^+) and (OH^-) products must equal 1.0×10^{-14}. Therefore, three situations are possible:

- A neutral solution, where $(H^+) = (OH^-)$
- An acidic solution, where $(H^+) > (OH^-)$
- A basic solution, where $(H^+) < (OH^-)$

The **pH meter** (Figure 11.12) is simply a voltmeter capable of measuring small differences in electrical potential between the responsive and reference cells, which are located in one tube called an **electrode**. The cells are separated by a ceramic or fiber junction. The **reference cell** is a closed loop and contains an electrolyte solution, usually silver chloride (AgCl). The **responsive cell** is an open loop (has a hole) and is filled with the filling solution. The filling solution is usually potassium chloride (KCl), which has a constant electrical potential. When the electrode is placed in a solution with a different potential from the reference electrode, an electrical potential occurs and is measured by the voltmeter. The pH meter converts the voltage into pH values.

Potentiometry is a temperature-related process (*pH increases with a decrease in temperature*). The potentiometric process is a hydrolysis reaction that speeds up at higher temperatures, causing the pH to decrease. For example, pure water has a pH of 7.0 at 25°C, but at 50°C it decreases to 6.6 and at 100°C it drops as low as 6.1. If the pH of a juice sample is 8.2 at the temperature of second carbonation (85°C), the pH value increases to about 9.2 when the sample is cooled to 20°C. Therefore, pH measurements can be compared *only on tests carried out at the same temperature*.



Figure 11.12 A pH meter

NOTES ON POTENTIOMETRY

- **pH meters** are calibrated and verified using standard buffer solutions. **Commercially prepared buffer solutions** with values of 4, 7, and 10 are recommended for checking of the pH meter. Buffer solutions with different pH values have different colors, which makes the calibration easier.
- Before a pH measurement can be taken in the lab, the samples must be cooled to the standard temperature (20°C).
- The **calibration** of the pH meter is important because the electrical potential of electrodes varies over time; therefore, the pH meter should be **calibrated** (standardized) with a buffer solution at the end of each shift. The calibration is usually performed using 4 and 10 buffer solutions. A buffer solution is prepared by dissolving a weak acid and a salt of its base in water, such as acetic acid and sodium acetate.
- The **calibration procedure** of the pH meter depends on the brand. Each comes with a manual that explains the procedure.
- **Samples** heavier than 50% *DS* should be double-diluted with distilled water before the pH measurement.
- **Newer electrodes** contain a built-in temperature probe offered by the electrode suppliers as pH/ATC combination electrodes.
- **Junction clogging** in the reference electrode is a common problem with combination electrodes, creating wrong results in pH meter operations. If this occurs, you should replace the electrode filling solution (KCl solution) by applying the refill to the filling hole of the electrode with a plastic dropper. Then, you should soak the electrode in distilled water for few hours.

- **Flushable-junction combination electrodes** prevent junction clogging. These electrodes have no ceramic or fiber junction to clog.
- An **electrode's slow response** can be corrected by immersing the electrode in a 10% HCl and rinsing it several times with distilled water.
- **Potentiometric titration** is a titration method based on the addition of a titrant of known concentration to a solution of unknown concentration, until a certain pH value is obtained. Then the unknown concentration can be related to the titrant concentration and used volume.
- **Indicator pH papers** are often used in the factory to measure the pH of juices from the purification station. These indicators change color when used in samples with different pH values. The operator then matches dominant color of the paper to the color of the blocks on the tester box.

SPECTROPHOTOMETER

HISTORY

In 1861 K. Stammer was the first to publish a description of a colorimeter (spectrophotometer). The original model went under different modifications until today's spectrophotometers that digitaly displays the result of color measurement in numbers and independent of the observer's judgment.

The spectrophotometer (colorimeter) measures the color in a solution sample. The technique used by the spectrophotometer is called **spectrophotometry**, which is based on the intensity of light absorbed (or transmitted) when a **spectrum** (wave) of the light passes through a colored solution (light-absorbing solution). A simple spectrophotometer (see Figures 11.13 and 11.14) consists of the following:

- A sample cell
- A sample-cell holder
- A bulb that emits wavelengths
- A prism that creates a spectrum of the light
- A device that selects the appropriate wavelength of the light
- A detector that generates an electrical current proportional to the light intensity
- A display that shows the received signals in transmittance or absorbance in numerical values

Normal light consists of a few colors that can be separated into component colors by a prism (or a diffraction grating) to form a **spectrum** (ray) of the light, which is a light with a narrow-band width. In a spectrophotometer, a spectrum of the light strikes a solution sample. As the light passes through the sample, part of its intensity is absorbed by the sample (*the higher the color of the sample, the more of the spectrum of the light is absorbed by it*). Therefore, the intensity of the light leaving the sample (I) is less than the entering intensity (I_0). The ratio of intensities is called the **transmittance** (T):

$$T = \frac{I}{I_0} \tag{11.6}$$

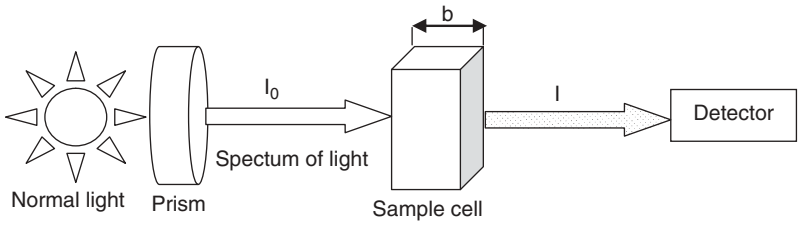


Figure 11.13 Basic parts of a spectrophotometer

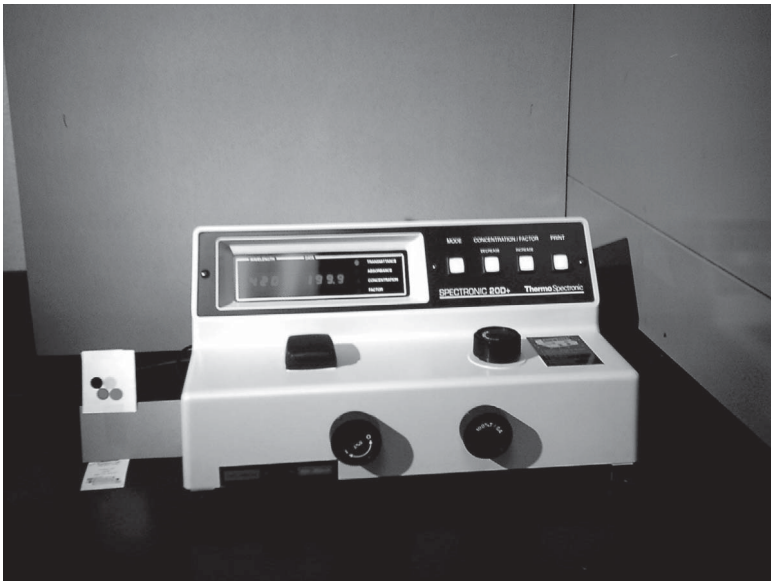


Figure 11.14 A spectrophotometer

Or, expressed as a percentage, so it can be written as

$$\%\, T = \frac{I}{I_0} \times 100$$

The detector of the spectrophotometer is calibrated before measuring T of the sample. Typically, the calibration is achieved by assigning distilled water a value of 100 T (because absorbance of pure water is 0) and a value of 0 for T is assigned by blocking the detector so that no light can enter. In fact, the spectrophotometer compares the ratio of the intensity of the transmitted light (unabsorbed light) of a sample to the transmitted light of a **blank** or reference (distilled water):

$$T = \frac{I_{\text{Sample}}}{I_{\text{Black}}} \tag{11.7}$$

or

$$\%\,T = \frac{I_{\text{Sample}}}{I_{\text{Black}}} \times 100$$

The reciprocal (inverse) log of T is called **absorbance** (A), which is a measure of the part of light absorbed by the sample:

$$A = \log \frac{1}{\%\,T} \tag{11.8}$$

The **color** (C) of a solution can be calculated using the Lambert-Beer law:

$$C = \frac{A}{b \times c} \tag{11.9}$$

b Sample-cell thickness (light path length), the distance of the light traveling through the sample (cm)
c Concentration of sample (% m/v)

A closer look at Eq. (11.9) shows a linear relationship between C and A as long as we use the same concentrated sample, size cell, and wavelength for all our measurements.

When expressing the path length in mm (millimeter), the formula becomes

$$C = 10\frac{A}{b \times c} \tag{11.10}$$

The concentration (c) of the sample when using the dry substance (DS) and the density (d) of the sample is given by

$$c = \frac{DS \times d}{100} \tag{11.11}$$

Substituting c into Eq. (11.9) obtains

$$C = 1000\frac{A}{b \times DS \times d} \tag{11.12}$$

In the sugar industry, the value of C (measured at 420 nm) multiplied by 1000 is reported as ICUMSA-420 color (IU). Therefore, the color equation becomes

$$C = 10^6\frac{A}{b \times DS \times d} \tag{11.13}$$

When using transmitance, Eq. (11.12) should be derived based on the relation between A and T. Absorbance is the inverse log of T.

$$A = \log \frac{1}{\%\,T} = -\log \%\,T = -(\log T - \log 100) = -\log T + 2 = 2 - \log T \tag{11.14}$$

$$C = 10^6\frac{2 - \log T}{b \times DS \times d} \tag{11.15}$$

For measuring the color of a sample, A or T is taken from the spectrophotometer reading and the DS of the sample is measured on the refractometer. The density of the sample can be taken from the DS density table (Table A.1 of the Appendix).

The Fast Way to Calculate Color

Among other variables, Eq. (11.15) is density (**d**) dependent. In the lab, we measure the *DS* of the sample and find its *d* from the *DS* density table (Table A.1 of the Appendix) to calculate color of the sample. This is a time-consuming procedure. In the tables of the Appendix and in the Basics of Mathematics and Statistics section of Chapter 12, we found out that when two variables have a linear relationship (e.g., *d* and *DS*), their regression equation can be used to make the formula independent of the density of the solution being tested. The regression equation was given as

$$d = 0.0055 \times DS + 0.9714 \tag{11.16}$$

Therefore, the density of the sample in which the color has to be determined can be calculated by a combination of Eq. (11.16) with (11.13) or with (11.15):

$$C = 10^6 \frac{A}{b \times DS(0.0055 \times DS + 0.9714)} \tag{11.17}$$

$$C = 10^6 \frac{2 - \log T}{b \times DS(0.0055 \times DS + 0.9714)} \tag{11.18}$$

In using Eq. (11.17) or (11.18), we are no longer dependent on the *DS*-density table. We can put the color equations into the computer to calculate the color value of the samples. This saves a considerable amount of time.

With values of *A* (or *T*), *b*, and *DS* entered in separate cells of a Microsoft Excel spreadsheet, and the color formula—Eq. (11.17) or (11.18)—in another cell relating to other values, the computer can calculate the color value in a short time period. For example, *T* can be entered in cell A1, *b* in A2, and *DS* in A3, and the color equation obtained in any cell as it follows:

```
= 1000000*(2 – LOG(A1)/((A2*A3)*(0.0055*A3 + 0.9714))
```

EXAMPLE 2

Given:

DS of a sugar solution	50.2%
% *T*	90.4
Sample tube diameter	25.4 mm

Calculate:

Color (*C*) of the sugar solution (assume zero turbidity)

From the *DS* value in the *DS*-density table, the density is 1.23, so the color value is

$$C = 10^6 \frac{2 - \log T}{b \times RDS \times d} = 10^6 \frac{2 - \log 90.4}{25.4 \times 50.2 \times 1.23} = 10^6 \frac{2 - 1.956}{1275.1 \times 1.23} = 28$$

The result is the same if we use Eq. (11.18):

$$C = 10^6 \frac{2 - \log 90.4}{25.4 \times 50.2(0.0055 \times 50.2 + 0.9714)} = 10^6 \frac{0.044}{1275.1 \times 1.25} = 28$$

NOTES ON THE SPECTROPHOTOMETRY

- The measurement in a spectrophotometer is carried out at a specific wavelength. In ordinary spectrophotometers, a wavelength of 350 to 880 nm is usually used. In expensive instruments, the wavelength is extended into the violet region (wavelength below 350 nm).
- **ICUMSA units** (IU), called ICUMSA-420 color, is the color value measured by the spectrophotometer at 420 nm wavelengths. This method is used internationally in the sugar industry to express the color of in-process products (juices, syrups, and liquors), the final product (granulated-refined sugar), and by-product (molasses).
- **ICUMSA** (International Commission for Uniform Method of Sugar Analysis) recommends the color measurement of sugar and liquid sugar at two wavelengths of 420 and 720 nm (nanometer is equal to 10^{-9} m). An absorbance measurement at 720 corresponds to the turbidity of the solution, which is measured in the unfiltered sample. The measurement at 720 nm can be omitted for practical and routine analytical controls when the sample is filtered before the color measurement. Filtration removes fine substances that can affect the color of the solutions.
- A **dilution of the sample** is necessary when the sample is too dark to permit good color readings. Therefore, dark samples can be diluted volumetrically, and the results multiplied by the dilution factors.
- **Newer spectrophotometers** are equipped with a snap-in holder for holding both cuvets (square sample cells) and sample tubes (round sample cells).
- Cell **path length** (*b*) is the thickness of the solution through which the light is passing. It refers to the outside diameter of the cell (in round sample cells) or the width of the cell (in square sample cells). Sample tubes with a 10.0 mm or 25.4 mm (1 in.) path length are commonly used in sugar labs.
- Using a **sample cell** with a 25.4 mm path length, and a measurement at 420 nm wavelengths results in color units (in IU) the same as the **RBU** (reference basis units), which is the method used by the U.S. bottlers industry (National Soft Drink Association, NSDA).
- **Sample cells** should be cleaned both inside and out with distilled water after each test and dried with a soft cotton cloth. At the end of each shift, the sample cell should be cleaned with detergent, rinsed with distilled water, dried, and polished with a soft cotton cloth.
- **Sample cells** should be dried with a **soft cotton** cloth because paper tissue can scratch them, and cause them to produce a faulty result.
- The lower part of the sample cell should not be touched because fingerprints can change an optical response.
- Sample cells should be checked for scratches, and scratched cells should be eliminated.
- Sample and **blank** (reference) cells used for color measurement should be the same size and matched.
- The **sample cell** should be rinsed with a small portion of solution before taking a measurement of that solution.
- The accuracy of the spectrophotometer should be checked weekly by using a standard set blocks. These blocks allow the calibration of the wavelength and absorbance for almost any spectrophotometer. The result of the reading against standard block sets should be in the ±3% range of the standard value. With faulty calibration results, the instrument should be adjusted according to the manufacturer's instruction.
- **Automatic zero-transmittance**, a device in newer models of spectrophotometers, can zero the instrument without the need of a switch.
- **Spectrophotometry** (colorimetry) can determine the concentration of unknown samples by comparing the color of the sample with a set of standard solutions. This is possible if the sample develops a stable color with another substance. This method is known as **spectral analysis** and is used mainly in sugar labs for environmental analyses such as determining the phosphorous in wastewater.

CONDUCTOMETER

The **conductometer** (conductivity meter) measures **ash** (soluble inorganic compounds) content in a solution sample. The technique used by the conductometer is called **conductometry**, which is based on the electrical conductance of a sample when electric current carried out through the sample (*the higher the conductance, the higher is the concentration of the ions in the sample*). Solutions carry electric current with different speeds depending on their strength of ionization in solution. **Ionic substances** (electrolytes) such as salt (NaCl) produce large numbers of ions in solution, forming a *conducting solution* that conducts electric current easily. **Nonionic substances** (nonelectrolytes) such as sucrose ($C_{12}H_{22}O_{11}$) do not ionize in solution, so they do not produce ions in solution, forming a *nonconducting solution*.

Pure water is a nonelectrolyte because there is no substance to conduct electric current. However, when an ionic substance is added to water, it conducts electric current much easier. The reason is that positive and negative ions are formed, which allows for the passage of the current. The positive ions (cations) in the solution migrate to the negative electrode (cathode), while the negative ions (anions) are attracted to the positive electrode (anode). Such migrations provide the necessary contact between the electrodes.

Electrical conductivity of impure sucrose solutions is much higher than that of pure sucrose solutions due to the presence of **ash substances** (soluble inorganic compounds). Therefore, the conductance created in the impure sucrose solutions comes from the ash present in the sample. Based on this explanation, the ash content of the solution can be measured indirectly by conductivity. Ash determined by conductivity is called **conductivity ash**.

The conductivity meter recognizes the electrical current, and compares the **electrical conductance** (the reciprocal of electrical resistance) of a sample to a calibrated **cell** (electrode). When the conductivity cell is placed in an ionic solution, an electrical circuit is formed across the cell. This results in a conductivity value that can be read on the display of the conductivity meter. Each substance has its own degree of conductance; the conductance is proportionally related to the concentration of that substance in the solution.

The following factors affect the degree of conductance (K):

- Proportionally to the cross-sectional area (A) of the electrodes
- Inverse-proportionally to the distance (D) between electrodes

$$K = \frac{1}{R} = K_C \frac{A}{D} \tag{11.19}$$

Conductance is the reciprocal of resistance (**R**), and K_C is the cell constant. K_C is determined by measuring the resistance of a standard solution of known specific resistance (see the Notes). **Specific conductance** of a solution is defined as the conductance of a column of liquid 1 cm long and having a cross-sectional area of 1 cm^2. The unit for conductivity is siemens (S) or microsiemens (μS) and the unit for K_C is 1/cm.

In sugar labs, the ash content of in-process products and sugar is measured by conductivity due to the following two good reasons:

- The gravimetric method for ash determination takes a long time
- The results of the conductivity method are close to those found by gravimetric method

The **gravimetric method** of ash determination refers to a **sulfated-ash method**. In this method, all volatile materials, such as Cl, NO_3, and CO_2, are replaced by SO_4 using concentrated sulfuric acid and heated in a muffle furnace until all the carbon is burned. The determination of ash content by conductivity offers a fast and environmentally friendly alternative to the sulfated-ash method.

NOTES ON THE CONDUCTOMETRY

- **The term ash (soluble inorganic compounds) is derived from determination of the inorganic compounds by heating the sample in the furnace to burn the organic compounds. The residue (called ash) contains the oxides and sulfates of K, Na, Ca, and Mg cations.**
- **Inorganic cations (e.g., sodium, potassium and calcium), inorganic anions (e.g., nitrate, chloride, and carbonate), insoluble materials (e.g., filter aid and sand), metals (e.g., filings and welding slag), and organic salts (e.g., calcium carboxylates) in sugar are examples of ash content. Minerals that are in the sugarbeet, added in the process, or eroded from processing equipment may become part of ash.**
- **Ash without any qualifying expression is referred to conductivity ash.**
- **Conductivity cells are obtained commercially with different cell constants (usually from 0.1 to 10.0 cm^{-1}). The conductivity cells are an immersion-type similar to pH electrodes and usually are platinized.**
- **Distilled or deionized (DI) water with low specific conductance should be used to prepare solutions for conductivity measurement.**
- **Pure sucrose solutions, boiler feedwater, and distilled water have very low conductance (around 200 μS/cm), so you should measure their conductivity using an electrode with a small cell constant (0.1 to 1.0 cm^{-1}).**
- **The accuracy of the conductivity cell should be checked once in a while with a calibration solution such as a potassium chloride (KCl) solution with a specified concentration (molarity) and conductance. The calibration solution can be purchased from laboratory chemical suppliers. To prepare it in house, use the following procedure:**
 1. **Dry about 1 g of KCl at 105°C for 2 hours and cool it in a desicator.**
 2. **Prepare a 0.01*N* KCl solution by dissolving 0.745 g of dry KCl in a 1000-ml flask with distilled water. (The molecular mass of KCl is 74.5 g/mole.) This solution has a conductivity of 1278 μS/cm at 20°C.**
 3. **Keep the flask in a water bath for 30 minutes at the standard temperature (20°C).**
 4. **Transfer about 100 ml of the solution into a beaker and determine its conductivity.**
 5. **Calculate the cell constant (K_C) as follows:**

$$K_C = \frac{1278}{\text{Conductivity reading}}$$

For example, if the conductivity reading is measured 1447 μS/cm, the cell constant will be

$$K_C = \frac{1278}{\text{Conductivity reading}} = \frac{1278}{1447.5} = 0.88\ 1/\text{cm}$$

CHAPTER
11

SECTION *3*

LABORATORY REAGENTS

SUBSECTIONS

- Deionized-Water Preparation
- Reagent Preparation

Knowing how to prepare purified water is needed for the preparation of the lab reagents, volumetric dilutions of samples, and other routine lab practices. So we first explain how to prepare deionized-water (DI water), known to chemists as distilled water or deionized water (**DI water**).

DEIONIZED-WATER PREPARATION

The characteristic for specifying the quality of purified water is its **conductance** (the ability of carrying electric current). The average city water has high conductivity because it contains dissolved suspended solids. Therefore, it is characterized as **hard water** (water with a considerable amount of calcium and magnesium ions). If bicarbonate (HCO_3) ions are present in hard water, boiling the water causes a precipitate of calcium carbonate (CO_3Ca), which is not desired. Hard water cannot be used untreated in the lab, and it is usually deionized by one of the following techniques:

- Distillation
- Ion-exchange resin
- Reverse-osmosis process

The **distillation process** can deionize water. In distillation, city water is boiled and the steam collected and condensed. Water purified by distillation is called **distilled water.** In the lab, an electrically heated still is usually used for water distillation. High-quality distilled water has a specific conductance of 1 μS/cm, which corresponds to about 0.5 ppm of dissolved solids.

The **ion-exchange resin** (simply resin) is the most common method used in today's labs for preparation of DI water. The process is similar to water softening in households, which involves the removal of the hardness from hard water by resin. To prepare DI water, city water is passed through a column filled with resin. Resin consists of tiny, insoluble, porous particles capable of attracting cations and anions, depending on its functional group (see Chapter 5).

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In this method, cations that cause water hardness (Ca and Mg) are exchanged for similarly charged sodium ions attached to the resin particles. At the same time, sodium cations leave the resin and dissolve in the water. Therefore, undesirable hard-water ions are exchanged for sodium ions. The resin particles should be **regenerated** when they get exhausted (lose exchanging capacity). For regeneration, salt water, containing Na ions, is pumped through the resin to replace the Ca and Mg ions and bring the resin to its original exchanging capacity (see Chapter 5).

To prepare better quality DI water, the **mixed-bed ion-exchange** system is used in which strong cation and anion resins are used separately in two different columns to take care of both the cations and anions existing in the water.

The advantage of the deionization system over the distillation system is its capacity for producing sufficient quantities of purified water for lab use. Its disadvantages are as follows:

- The deionization method does not remove bacteria, so the system can get contaminated
- The deionization method adds organic substances to the water

(Note: If system gets contaminated, the DI water produced is not suitable for either BOD or COD analyses.)

In the **reverse osmosis** process, water is deionized by applying a pressure across a semipermeable membrane. The reverse-osmosis technology (discussed in Section 1 of Chapter 12) separates the dissolved solids out of the water, in average, by about 98%.

Reagent-grade water is very high quality purified water that has been first pretreated by resin (or reverse osmosis) and distillation. It is then passed through an activated carbon cartridge (column). Reagent-grade water has 100 to 300 µg/L or ppb (part per billion) of dissolved solids. It is used for procedures such as **high-performance liquid chromatography** (HPLC).

NOTES

- **Deionized water (DI water) prepared by exchange resin is used in the preparation of reagents and sample dilutions explained in this book.**
- **Lab purified water can be stored in containers or distributed throughout the lab by the plumbing system. For storing water in containers, polyethylene plastic bottles are recommended because they do not cause organic contamination.**
- **Always read the label of chemical bottles twice before using any reagents. It is easy to misread a label.**
- **A chemical-solution calculator, offered by instrument manufacturers, can help chemists calculate some chemical equations, such as the amounts of solute and solvent in mass and volume fractions.**

REAGENT PREPARATION

Reagent preparation is one of the most important jobs in any sugar laboratory. The reagents used in sugar labs are prepared so that their concentrations are known.

The general steps in preparing reagents are as follows:

- Find out the molecular mass of the chemicals
- Calculate the amount of chemicals needed

- Measure the right amount of chemicals
- Dissolve the chemicals in DI water
- Adjust it to the exact volume
- Standardize it if required

In this section, reagents are recognized by a **number**. For example, **Reagent 1** is a 20% solution of aluminum chloride hexahydrate ($AlCl_3.6H_2O$). The following are some general notes that can help you prepare lab reagents.

NOTES

- **Prepare the solution slightly overstrength so that you can add more water at the time of standardization. Preparing an understrength solution is not advised because the amount of the stock needed for an adjustment might be very small and therefore difficult to measure (see Example 33 in Chapter 12).**
- **Laboratory chemicals are manufactured with different degrees of purity, as follows:**
 Reagent grade is usually used for reagent preparation.
 Chromatographic grade is used for HPLC.
- **Buffer solutions, for pH meter calibration, can be purchased from lab-chemical suppliers. Therefore, no procedure is provided in this text for the preparation of these solutions. Buffer solutions with pH values of 4, 7, and 10 are usually used for the pH meter calibration.**
- **For reagent preparation, the laboratory should be equipped with sufficient numbers of different-sized lab glassware such as beakers, volumetric flasks, pipettes, burettes, and graduated cylinders (see Figure 11.15).**
- **An analytical balance (see Figure 11.16) with a readability of 0.0001 g and a capacity up to 200 g is one of the most important instruments for reagent preparation.**



Figure 11.15 Laboratory glassware

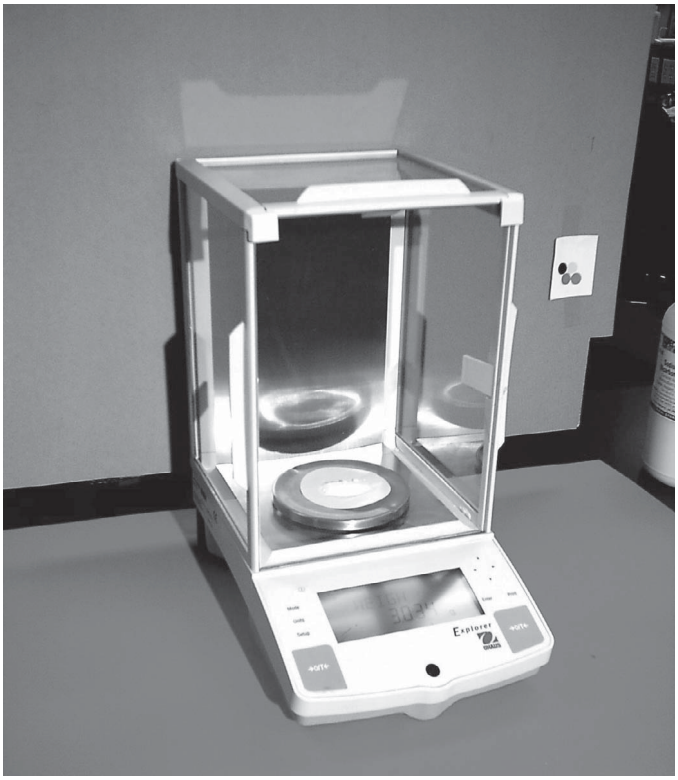


Figure 11.16 An analytical balance

Reagent 1 (20% Aluminum Chloride.6H_2O Solution)

Reagent 1 is used to determine the amount of sugar in cossettes and diffusion juice. To prepare this solution, dissolve 200 g aluminum chloride hexahydrate ($AlCl_3.6H_2O$) in about 500 ml DI water in a 1-L volumetric flask. Adjust it to the mark of the flask with DI water at the standard temperature (20°C) and mix it well.

Reagent 2 (20% Ammonium Hydroxide Solution)

Reagent 2 is used with **Reagent 1** to determine the amount of sugar in cossettes and diffusion juice. To prepare 20% (by volume) ammonium hydroxide solution, add 200 ml of ammonium hydroxide (NH_4OH) to a 1-L volumetric flask. Adjust the volume to the mark of the flask with DI water and mix it well. Use ammonium hydroxide 28% assay (as NH_3).

NOTES

- **There is no need to standardize reagents 1 and 2.**
- **Make several liters of these reagents at a time in a larger bottle (e.g., a 10-L bottle) because they are used frequently.**

Reagent 3 (Aluminum Chloride.6H_2O, Powder)

Reagent 3 is crystalline aluminum chloride hexahydrate ($AlCl_3$. $6H_2O$).

Reagent 4 (Calcium Hydroxide, Powder)

Reagent 4 is calcium hydroxide powder [$Ca(OH)_2$].

Reagent 5 (Phenolphthalein Indicator)

Regent 5 is phenolphthalein indicator. For preparation, dissolve 5 g of phenolphthalein in 500 ml of methanol (CH_3OH) and mix to 1 L with DI water. Add 1 to 2 ml diluted sodium hydroxide solution (about 10% solution) until a faint **pink** color appears.

Reagent 6 (0.036 *N* Sulfuric Acid Solution)

Reagent 6 is used for alkalinity tests of first- and second-carb juice. Each ml of this acid is equivalent to 1 mg CaO in a titration test. For preparation, add 1 ml of H_2SO_4 (96.0% assay with density of 1.84 g/ml) to about 500 ml of DI water, fill up to 1 L with DI water, and mix it very well.

You can prepare several liters of this solution at a time in a larger bottle (e.g., a 10-L bottle) because this reagent is used frequently.

The amount of the stock can be calculated by equivalent mass formula given in Chapter 12. The equivalent mass (*Eq*) is based on the molecular mass (*MM*) of the substance (the *MM* of H_2SO_4 is 98) and its number of charges (N_C; the N_C of H_2SO_4 is 2):

$$Eq(H_2SO_4) = \frac{98}{2} = 49\,\text{g equivalent}$$

$$Eq(CaO) = \frac{56}{2} = 28\,\text{g equivalent}$$

You can calculate the amount of H_2SO_4 needed to prepare 1 liter of 0.036 **normal** (*N*) solution by using the following equation:

$$\text{Mass}_{\text{Solute}} = N \times Eq_{\text{Solute}} \times \text{L}_{\text{Solution}}$$

$$\text{Mass}_{\text{Solute}} = 0.036 \times 49 \times 1 = 1.76\,\text{g}$$

The calculation can be derived also from the ratio of the equivalent mass of H_2SO_4 to CaO:

$$\frac{Eq_{H_2SO_4}}{Eq_{CaO}} = \frac{49}{28} = 1.75\,\text{g}$$

Because the assay (concentration) of the acid is 96.0%, the correct weight of the acid will be

$$\text{Mass} = 1.76 \times \frac{100}{96.0} = 1.84\,\text{g}$$

or

$$\text{Volume} = \frac{d}{\text{Mass}} = \frac{1.84\,(\text{g/mL})}{1.84\,(\text{g})} = 1.0\,\text{ml}$$

In titration calculations, each ml of prepared sulfuric acid solution is equivalent to 1 mg CaO (1 g per liter) or 1000 mg/L, which is 1000 ppm.

For **standardization**, titrate 20 ml of 0.036 *N* Na_2CO_3 solution (see the next paragraph for the preparation instructions) with the prepared sulfuric-acid solution using **Reagent 7** (special indicator) as an indicator. This should equal to 20 ml of acid solution. Adjust the strength of the acid if it is not correct.

To prepare 0.036 *N* Na_2CO_3, dissolve 1.9 g of dry Na_2CO_3 in 1 L of DI water. First, dry a little more than the needed amount of the sodium carbonate in the oven at 110°C for 2 hours, and then cool it in the desicator for 10 minutes.

The amount of Na_2CO_3 can be calculated by equivalent mass formula given in Chapter 12. The equivalent mass (*Eq*) is based on the **molecular mass** (*MM*) of the substance (the *MM* of Na_2CO_3 is 106) and its number of charges (N_C; the N_C of Na_2CO_3 is 2). The calculations follow:

$$Eq_{Na_2CO_3} = \frac{106}{2} = 53\,\text{g equivalent}$$

$$\text{Mass}_{\text{Solute}} = N \times Eq_{\text{Solute}} \times \text{L}_{\text{Solution}} = 0.036 \times 53 \times 1 = 1.9\,\text{g}$$

The amount of Na_2CO_3 can be also calculated from the equivalent mass (*Eq*) ratio of Na_2CO_3 to CaO:

$$\frac{Eq_{Na_2CO_3}}{Eq_{CaO}} = \frac{53}{28} = 1.9\,\text{g}$$

NOTES

- **If an adjustment needed after the titration for standardization, calculate the additional amount of the acid or water according to Examples 33 and 34 given in Chapter 12.**
- **Prepare the solution slightly overstrength so that you need to add more water, not acid. Otherwise, the amount of stock needed for adjustment might be too small to measure accurately.**

Reagent 7 (Special Indicator)

Reagent 7 is used as an indicator. To prepare this reagent, dissolve 0.02 g methyl red and 0.10 g bromcresol green in 100 ml of ethyl alcohol (ethanol).

Reagent 8 (Hardness Buffer Solution)

Reagent 8 is used as a buffer solution to determine **hardness** (limesalt) in different samples. For preparation, add 57 ml ammonium hydroxide (NH_4OH) to 7 g ammonium chloride (NH_4Cl), and dilute to 100 ml with DI water in a volumetric flask.

Reagent 9 (Hardness Indicator)

Reagent 9 is used as an indicator to determine hardness. At a pH of 10, the color of this indicator changes from **wine red** to **blue**. For preparation, mix 1.5 g hydroxynaphtol blue with 100 g of crystalline potassium chloride (KCl).

NOTE

Reagents 8 and 9 can be purchased from chemical suppliers. Chemical suppliers categorize Reagent 8 as a hardness buffer and Reagent 9 as a hardness indicator, which is made from eriochrome black.

Reagent 10 (Hardness Titrant)

Reagent 10 is used as the titrant to determine hardness. For preparation, dissolve 6.70 g of ethylendiamine tetraacetic acid dihydrate (EDTA.2H_2O, later simply **EDTA**) in about 500 ml of DI water and fill up the volume to 1 L with DI water. (Note that EDTA dissolves slowly.)

You can prepare several liters at a time in a larger bottle (e.g., a 10-L bottle) because this reagent is used frequently. For titration calculations, remember that 1 ml of this reagent is equal to 1 mg CaO.

The amount of EDTA can be calculated by equivalent mass formula given in Chapter 12. The equivalent mass (*Eq*) is based on the molecular mass (*MM*) of the substance (the *MM* of EDTA.2H_2O is 372) and its number of charges (N_C; the N_C of EDTA.2H_2O is 2):

$$Eq_{\text{EDTA}} = \frac{372}{2} = 186\,\text{g equivalent}$$

$$Eq_{\text{CaO}} = \frac{56}{2} = 28\,\text{g equivalent}$$

You can calculate the amount of EDTA needed to prepare 1 L of 0.036 normal solution by using the following equation:

$$\text{Mass} = N \times Eq \times \text{L}_{\text{Solution}} = 0.036 \times 186 \times 1 = 6.7\,\text{g}$$

The amount of EDTA can be also calculated from the equivalent mass (*Eq*) ratio of EDTA to CaO:

$$\frac{Eq_{\text{EDTA}}}{Eq_{\text{CaO}}} = \frac{186}{28} = 6.65\,\text{g}$$

In titration calculations, each ml of prepared EDTA solution equals 1 mg CaO (1 g/L) because

$$\text{Equivalent} = \frac{\text{Mass}}{\text{Equivalent mass}} = \frac{1.0}{28.0} = 0.036$$

For standardization:

- Titrate 20 ml of 0.036 *N* $CaCO_3$ solution (see the next paragraph for preparation instruction) with the prepared EDTA solution using 2 ml of **Reagent 8** (hardness buffer solution) for a pH adjustment and 3 shakes of **Reagent 9** (hardness indicator).
- Stir to dissolve the indicator.
- Titrate to the first color change, and then proceed slowly (drop by drop) to the **blue** endpoint.
- The titration should need 20 ml of EDTA solution. Adjust to the right strength if it is not correct.

To prepare 0.036 *N* of $CaCO_3$ solution, dissolve 1.80 g of the dry $CaCO_3$ and dilute to 1 L with DI water. First, dry a little more than the needed amount of calcium carbonate in the oven at 110°C for 2 hours, and then cool it in the desicator for 10 minutes. Next, weigh 1.80 g of the dry calcium carbonate and dilute it in a little diluted hydrochloric acid (about 10 ml of 1 : 1 HCl).

$$Eq_{CaCO_3} = \frac{100}{2} = 50\,\text{g equivalent}$$

$$\text{Mass} = N \times Eq \times L_{\text{Solution}} = 0.036 \times 50 \times 1 = 1.8$$

or

$$\frac{Eq_{CaCO_3}}{Eq_{CaO}} = \frac{50}{28} = 1.8\,\text{g}$$

If an adjustment is needed after titration, calculate the additional amount of acid or water according to Example 33 or 34 given in Chapter 12.

NOTES

- **EDTA is a chelate, a substance that can form a complex compound with calcium and magnesium). It is therefore used as a reagent to analyze solutions for hardness.**
- **EDTA is used also as a scavenger to remove calcium salts from factory sugar juices and to clean hardness from evaporators. (This way of evaporator cleaning is not common in sugar factories because of the high expense.) EDTA is also found in some products, such as soda, beer, soap, and cleaners to bind to trace metal ions that would otherwise produce unwanted precipitates.**

Reagent 11 (2.5% Aluminum Chloride Solution)

Reagent 11 is used to determine the amount of sugar in **carbonation lime** (limecake). To prepare a 2.5% m/v of this reagent, dissolve 25.0 g aluminum chloride ($AlCl_3.6H_2O$) in about 500 ml DI water in a 1-L volumetric flask. Adjust the volume to the mark of the flask with more DI water, and mix well.

Reagent 12 (1.78 *N* Nitric Acid Solution)

Reagent 12 is called Steffen nitric acid, and it is used to determine the amount of CaO in carbonation-lime residue (limecake). (For calculations, note that 1 ml of this reagent is equal to 50 mg CaO.) For preparation, dilute 112 ml of nitric acid (usually 70.6% assay acid with specific gravity of 1.42 is used) to a total volume of 1 L with DI water. You may prepare the solution slightly stronger so that you can add more water, not acid, at the time of standardization.

The amount of the required stock is based on the following calculations:

$$Eq_{HNO_3} = \frac{63}{1} = 63\,\text{g equivalent}$$

$$Eq_{CaO} = \frac{56}{2} = 28\,\text{g equivalent}$$

$$\text{Mass}_{\text{Solute}} = N \times Eq_{\text{Solute}} \times L_{\text{Solution}}$$

$$\text{Mass}_{\text{Solute}} = 1.78 \times 63 \times 1 = 112\,\text{g}$$

Because the assay of the acid is 70.6%, the correct acid weight will be

$$\text{Mass} = 112 \times \frac{100}{70.6} = 159\,\text{g}$$

or

$$\text{Volume} = \frac{d}{\text{Mass}} = \frac{159\,(\text{g/mL})}{1.42\,(\text{g})} = 112\,\text{mL}$$

In titration calculations, each ml of the prepared acid equals 50 mg CaO per ml (50 g/L) according to the following equations:

$$\frac{Eq_{HNO_3}}{Eq_{CaO}} = \frac{63}{28} = 2.25\,\text{g}$$

$$\frac{112}{2.25} = 49.80 \equiv 50\,\text{g/L}$$

For standardization, titrate 20 ml of 1.78 *N* Na_2CO_3 solution (see the next paragraph for preparation instruction) with a prepared acid solution, using **Reagent 7** (special indicator) as an indicator. This should equal 20 ml of nitric acid solution. Adjust the acid strength if it is not correct.

To prepare 1.78 *N* Na_2CO_3, dissolve 94.0 g of dry Na_2CO_3 in a little diluted hydrochloric acid (about 10 ml of 1 : 1 HCl), and dilute to 1 L with DI water. Before weighing, dry the calcium carbonate in an oven.

$$Eq_{Na_2CO_3} = \frac{106}{2} = 53\,\text{g equivalent}$$

$$\text{Mass} = N \times Eq \times L_{\text{Solution}} = 1.78 \times 53 \times 1 = 94\,\text{g}$$

If an adjustment is needed after the titration, calculate the additional amount of the acid or water according to Example 33 or 34 in Chapter 12.

Reagent 13 (1.78 *N* Sodium Hydroxide Solution)

Reagent 13 is called Steffen sodium hydroxide, and it is used to determine the amount of CaO in carbonation lime. For preparation, dissolve 71.2 g of sodium hydroxide in about 0.5 L of DI water and make up the volume to 1 L.

$$\text{Mass} = N \times Eq_{NaOH} \times L_{\text{Solution}} = 1.78 \times 40 \times 1 = 71.2\,\text{g}$$

For standardization, titrate 20 ml of 1.78 *N* HNO_3 solution with a prepared sodium hydroxide solution using **Reagent 5** (phenolphthalein) as an indicator. This should equal 20 ml of sodium hydroxide solution. Adjust to the right strength if it is not correct.

Reagent 14 (0.36 *N* Sulfuric Acid Solution)

Reagent 14 is used to determine the amount of CaO in different carbonation juices. (For titration calculations, note that 1 ml of this reagent is equal to 10 mg CaO.) For preparation, add 10 ml of H_2SO_4 (usually sulfuric acid with 96.0% assay and sp. gravity of 1.84 is used) to about 500 ml of DI water, bring the volume with DI water to 1 L, and mix very well.

For standardization, use 0.36 *N* sodium carbonate (Na_2CO_3) with **Reagent 7** (special indicator) as an indicator. For Na_2CO_3 preparation, dissolve exactly 19.1 g of dry sodium carbonate in 1 L of DI water:

$$Eq_{Na_2CO_3} = \frac{106}{2} = 53\,\text{g equivalent}$$

$$\text{Mass} = N \times Eq \times L_{\text{Solution}} = 0.36 \times 53 \times 1 = 19.1\,\text{g/L}$$

If an adjustment is needed after standardization titration, calculate the additional amount of the acid or water according to Examples 33 and 34 in Chapter 12.

Reagent 15 (0.36 *N* Sodium Hydroxide Solution)

Reagent 15 is used to determine the amount of CaO in different carbonation juices. For preparation, dissolve ($40 \times 0.36 = 14.4$ g) sodium hydroxide in DI water, and fill up to 1 L. For standardization, titrate against 0.36 *N* sulfuric acid using phenolphthalein as the indicator.

Reagent 16 (Alpha-Naphtol Solution)

Reagent 16 is used to detect the presence of sugar traces in boiler feedwater. For preparation, make a 20% m/v alpha-naphtol in alcohol.

Reagent 17 (Concentrated Sulfuric Acid)

Reagent 17 is used to detect the presence of sugar traces in boiler feedwater. This reagent is a concentrated sulfuric acid with about 96% assay.

Reagent 18 (0.07 *N* Sodium Hydroxide Solution)

Reagent 18 is used to determine sulfuric acid strength in factories with **juice-softening** stations. In juice softening by weak-cation resin, sulfuric acid is used as a regenerant for the regeneration of resin. For preparation, dissolve 2.8 g sodium hydroxide in one liter of DI water, and mix very well.

$$\text{Mass} = N \times Eq \times L_{\text{Solution}} = 0.07 \times 40 \times 1 = 2.8\,\text{g}$$

Standardize the prepared reagent against 0.036 *N* sulfuric acid using **Reagent 7** (special indicator) or **Reagent 19** (methyl red) as an indicator. Each ml of base should use 2 ml of acid ($2 \times 0.036 = 0.07$). If the normality is not right, adjust to the right strength.

Reagent 19 (Methyl Red Indicator Solution)

Reagent 19 is an indicator. For preparation, dissolve 1 g of methyl red in 100 ml of methanol.

Reagent 20 (Methyl Orange Indicator Solution)

Reagent 20 is an indicator. For preparation, dissolve 1 g of methyl orange in 1 L of DI water.

CHAPTER
11

SECTION *4*

BEET-END METHODS OF ANALYSIS

SUBSECTIONS

- Sugar in Cossettes
- Diffusion-Juice Tests
- Sugar in Pulp
- Sugar in Press Water
- Alkalinity in First- and Second-Carb Juice
- Hardness in First–Carb and Thin Juice
- Carbonation-Lime Residue Tests
- CaO in Different Juices
- Moisture in Pulp
- Thin-Juice Tests
- Thick-Juice Tests
- Sugar Traces in Boiler Feedwater

SUGAR IN COSSETTES

Sampling interval: 2 times per shift

Cossettes are products of the slicing station. Sugar determination in cossettes is one of the most important tests that a sugar lab conducts. The calculation of a factory's yield is based on the **sugar entered** (the sugar in the cossettes multiplied by the mass of the beet cossettes processed) and the **sugar produced** (granulated-refined sugar as the end product of the factory). Therefore, extreme care must be taken throughout the cossettes test to ensure the integrity of the test.

1. Fill a pail (about 5 L in volume) with cossettes by grabbing cossettes from the middle, side, and top of the cossettes conveyor or from the slicers (equal amount from each slicer) free from steam or water.
2. Mix the sample well and immediately place enough cossettes into a food processor to fill it approximately three-quarters full.
3. Run the processor until no grinding is taking place. Open the container, wipe the cossettes from the sides, and pulse again.
4. Weigh 26.00 g of sample in a weighing dish.

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Day: Date: Sheet 1 (Beet-end)

	Sugar in coss.	Diffusion juice			Towers RDS		Sugar in pulp				pH										Alkalinity	
		RDS	Pol	Purity	Mid 1	Mid 2	Wet 1	Wet 2	Press pulp	Press water	Fresh water	Press water	Diff. juice	Mixed juice	Pre-carb	Inter. lime	1st carb	2nd carb	Thin juice	Thick juice	1st carb	2nd carb
7am																						
8																						
9																						
10																						
11																						
12pm																						
1																						
2																						
3																						
4																						
5																						
6																						
7																						
8																						
9																						
10																						
11																						
12am																						
1																						
2																						
3																						
4																						
5																						
6																						
Avg.																						

Figure 11.17 Beet-end lab sheet 1 (*factory with two tower diffusers)

Day: Date: Sheet 2 (Beet-end)

	Thin juice				Thick juice		Limesalts				Limecake			CO2	Pulp & Pellets					Lactic acid		
	% RDS	% Pol	% Purity	Color	% RDS	Color	Thin Juice Hard	Thin Juice S. Ash	Thin Juice Soft	Mol-asses	% Sugar	% CaO	% Moist.	% i n	% Moisture Wet	% Moisture Press	% Moisture Dry	% Moisture Pellets	%Sug. Pellets	Mid 1	Mid 2	Press water
7am																						
8																						
9																						
10																						
11																						
12pm																						
1																						
2																						
3																						
4																						

Figure 11.18 Beet-end lab sheet 2

5. Transfer into a 201-ml Kohlrausch flask (see the following Notes on marking the flask at the 201 ml point) by washing with DI water using a wash bottle. Use approximately 150 ml water for washing to leave room for the reagents added in further steps.
6. Place the flask in a water bath at 80°C for 30 minutes for **hot digestion** (see the Notes for **cold method**). During digestion, remove the flask every 10 minutes and shake it well.
7. Remove the flask from the water bath after 30 minutes and add 5 ml of **Reagent 1** (20% aluminum chloride solution) and 5 ml of **Reagent 2** (20% ammonium hydroxide solution).
8. Mix well by swirling.
9. Cool to standard temperature (20°C) in the water bath.
10. Wash the inside of the neck of the flask with DI water using a wash bottle and continue adding the water to below the neck of the flask.
11. If foam is on the surface, add 2 drops of ether to clear the foam.
12. Make the volume to the 201-ml line mark with DI water using a dropper. Pay attention that the bottom of the meniscus and the line correspond exactly.
13. Stopper the flask and mix it well by inverting and shaking.
14. Let it set for about one minute, allowing bubbles to escape.
15. Filter it into a clean beaker and return the first unclear portion (about 10 ml) of the filtrate (the liquid collected during filtration) back to the funnel or discard it. Use 18-cm Whatman #4 filter paper or the equivalent for filtration.
16. Rinse a polarimeter tube twice with clear sample and fill the tube again.
17. Set the polarimeter to 0, place the tube in the polarimeter, and record the reading to two decimal places when the reading is stabilized.

When a 100-mm polarimeter tube is used, the result is

$$\% \text{ Sugar} = \text{Polarimetric reading} \times 4$$

NOTES

- **In the analysis of the cossette sample, the marc (beet pulp) and, to lesser extent, the volume of the precipitate formed during clarification have to be considered because both affect the volume of the solution in the flask. These substances occupy part of the volume of the flask, so they raise the polarimetric reading. To solve this problem, it is assumed that the marc in 26.00 g of beet cossettes occupies a certain volume by which the volume of the flask must be increased to obtain the correct result. Sugar chemists have found that the pulp in 26.00 g (normal weight) of cossettes occupies approximately 1 ml. Therefore, a 100-ml flask should be graduated at 100.5 ml, and a 200-ml flask at 201 ml. (Note that 26.00 g of product in a 101-ml flask represents a normal solution and in a 201-ml flask represents a half-normal solution.)**
- **The 201-ml flask can be made from a 200-ml flask by filling it to the mark of the flask with DI water and then adding 1 ml more water to it at exactly 20°C temperature. Then remark the flask at 201 ml with a color fine-line felt-tip marker. These markers are resistant to both acid and alkaline, and are available at laboratory equipment suppliers.**
- **To test a cossette sample by cold digestion method, follow the preceding procedure up through step 3. Then, weigh 26.00 g of the sample on a weighing glassine paper and place it (with the paper) into a blender. Add 178.4 g of 0.3% $Al_2(SO_4)_3 \cdot 18H_2O$ solution to the blender and blend for 4 minutes. The rest of the procedure is the same as steps 15 through 17 of the preceding procedure (Thomas 1997).**

DIFFUSION-JUICE TESTS

Sampling interval: 2 times per shift

Diffusion juice is the product of the diffusion station, and is usually analyzed for dry substance, sugar, and pH. (Note: The presence of suspended solids in the diffusion juice affects the measurement results, particularly *RDS*.) Therefore, the diffusion juice should be filtered through a coarse filter (e.g., piece of centrifuge screen) before testing. Diffusion juice is analyzed for the following tests:

- **RDS**
- Sugar content
- pH

To test a **diffusion-juice** sample for ***RDS*** (refractometric dry substance or Brix), use the following procedure:

1. Cool the sample to the standard temperature (20°C).
2. Filter the sample through a coarse filter (e.g., a piece of centrifuge screen) to eliminate coarse particulate materials. Keep the sample for the sugar test.
3. Apply the sample on the refractometer and read the % *RDS*.

NOTE

If the measurement is carried out at a temperature other than the standard (20°C), the *RDS* should be corrected. For correction

For each degree above20°C, add 0.08 to the *RDS* reading
For each degree below 20°C, subtract 0.08 from the *RDS* reading

To test a **diffusion-juice** sample for **sugar content**, use the following procedure:

1. Weigh 26.00 g of filtered-cold sample (left from step 2 of the *RDS* test) in a weighing dish.
2. Transfer into a 200-ml Kohlrausch flask by washing with DI water using a wash bottle.
3. Add 5 ml of **Reagent 1** (20% aluminum chloride solution) and 5 ml of **Reagent 2** (20% ammonium hydroxide solution).
4. Mix well by swirling.
5. Add DI water from a wash bottle to below the neck of the flask.
6. If foam is on the surface, add 2 drops of ether to clear the foam.
7. Adjust the volume of the flask to its mark with DI water using a dropper. Pay attention that the bottom of the meniscus and the line correspond exactly.
8. Stopper the flask and mix it well by inverting and shaking.
9. Let it set for about one minute, allowing bubbles to escape.
10. Filter the solution into a clean beaker and return the first unclear portion (about 10 ml) of the filtrate back to the funnel or discard it. Use 18-cm Whatman #4 filter paper or equivalent for filtration.

11. Rinse a polarimeter tube twice with clear sample and fill the tube again.
12. Set the polarimeter to 0, place the tube in the polarimeter, and record the reading to **two decimal places** when the reading is stabilized.

When a 100-mm polarimeter tube is used, the result is

$$\% \text{ Sugar} = \text{Reading} \times 4$$

To calculate **purity** (*P*, the percentage of sugar in the total dry substances of a sample, divide the sugar by *RDS* and multiplying the result by 100:

$$\% P = \frac{\% \text{ Sugar}}{\% RDS} \times 100$$

NOTE

For higher accuracy, use the refractometer and polarimeter readings with two decimal places for the purity calculation. Then round up to one decimal place.

To test a **diffusion-juice** sample for **pH**, use the following procedure:

1. Cool the sample to the standard temperature (20°C).
2. Rinse the pH meter electrode and beaker with the cold sample.
3. Put sufficient sample in the beaker and immerse the electrode in the sample.
4. When the reading stabilizes, read the pH.

SUGAR IN PULP

Sampling interval: 2 times per shift

Sugar left in the pulp discharged from the diffuser is considered loss. Therefore, controlling the amount of sugar in the pulp during operation is very important. To determine the amount of sugar in wet, pressed, and dry pulp and pulp pellets, we use the polarimetric method.

Dry pulp and pulp pellet samples should be **ground**. Then weigh 10.0 g and proceed as follows:

1. Weigh samples as follows:

Wet pulp	20.0 g
Pressed pulp	20.0 g
Dry pulp	10.0 g
Pulp pellets	10.0 g

2. Transfer the weighed pulp into a blender.
3. Add 250 ml of cold **DI water**, 1/2 teaspoon of **Reagent 3** (aluminum chloride), and 1 teaspoon of **Reagent 4** (calcium hydroxide) to the blender.
4. Blend for 3 minutes.
5. Filter it into a clean beaker and return the first unclear portion (about 10 ml) of the filtrate back to the funnel.
6. Rinse a polarimeter tube twice with clear filtrate and fill the tube again.

7. Set the polarimeter to 0, place the tube in polarimeter, and record the reading when the polarimeter has stabilized.

When a 100-mm polarimeter tube is used, the result is

$$\% \text{ Sugar} = (\text{Reading} \times 0.52) \times \frac{250 - \% \text{ Moisture in sample}}{\text{Mass of sample}}$$

NOTE

Given the following approximate moisture contents, the formula for each sample becomes

Moisture in wet pulp = 85%	**% Sugar in wet pulp = Reading × 4.30**
Moisture in pressed pulp = 75%	**% Sugar in pressed pulp = Reading × 4.54**
Moisture in dry pulp = 10%	**% Sugar in dry pulp = Reading × 12.48**
Moisture in pellet pulp = 10%	**% Sugar in pulp pellets = Reading × 12.48**

SUGAR IN PRESS WATER

Sampling interval: 2 times per shift

Press water is produced by pressing the wet pulp with a pulp presses. For testing the wet pulp sample for sugar content, use the following procedure:

1. To about 200 ml of sample, add 1 level teaspoon of **Reagent 3** (aluminum chloride) and 1 level teaspoon of **Reagent 4** (calcium hydroxide).
2. Mixwell.
3. Filter the sample into a clean beaker and return the first unclear portion (about the first 10 ml) of the filtrate back to the funnel. Use 18-cm Whatman #4 filter paper or equivalent for filtration.
4. Let it cool down to the standard temperature (20°C).
5. Rinse a polarimeter tube with clear filtrate, fill the tube, and record the polarimetric reading when the polarimeter has stabilized.

When a 100-mm polarimeter tube is used, the result is

$$\% \text{ Sugar} = \text{Polarimetric reading} \times 0.52$$

ALKALINITY IN FIRST- AND SECOND-CARB JUICE

Sampling interval: 4 times per shift

Alkalinity is defined as the alkalinity produced by **alkaline-earth salts** (sodium, potassium, calcium, and magnesium salts). One of the most important tests that a sugar lab provides on a regular basis is the alkalinity test, which is usually performed on first- and second-carb filtered juice. The alkalinity test has been found to be a good indication of first- and second-carb juice quality. The test uses a standard acid solution (usually 0.036 N H_2SO_4) in the presence of a phenolphthalein indicator to a neutralization point (endpoint) of pH 8.

For testing a sample for alkalinity, proceed as follows:

1. Filter the sample.
2. Pipet 10 ml of the sample to a porcelain dish.
3. If the sample is too dark, dilute it with about 50 ml of DI water.
4. Add 3 drops of **Reagent 5** (phenolphthalein indicator) and titrate with **Reagent 6** (titrant, 0.036 N H_2SO_4 solution) until the **pink** color disappears entirely (endpoint of phenolphthalein equal to pH of 8.2).

Calculate the alkalinity (as grams CaO per 100 ml of sample) according to the following equation:

$$\text{Alkalinity} = \text{ml}_{\text{Titrant}} \times 0.01\,\text{g}/100\,\text{ml}$$

The preceding equation is based on the following calculation, by the fact that each ml of titrant (sulfuric acid solution) is equivalent to 1 mg or 0.001 g CaO:

$$\text{Alkalinity} = \text{ml}_{\text{Titrant}} \times 0.001 \times \frac{100}{\text{ml}_{\text{Sample}}}\,\text{g}/100\,\text{ml}$$

For 10 ml of sample,

$$\text{Alkalinity} = \text{ml}_{\text{Titrant}} \times 0.01\,\text{g}/100\,\text{ml}$$

NOTE

Back-titration is necessary if you miss the endpoint (the point in a titration at which the indicator changes color). At this point, the solution returns to a pink color. To back-titrate, add another 2 ml of juice to the dish to destroy the pink color, then re-titrate with the acid. Remember, the total volume of acid is equal to the total amount of acid in the titration and the back-titration step. The calculation is

$$\textbf{Alkalinity} = \textbf{ml}_{\text{Titrant}} \times \textbf{0.001} \times \frac{\textbf{100}}{\textbf{ml}_{\text{Sample}}} = \textbf{ml}_{\text{Titrant}} \times \textbf{0.001} \times \frac{\textbf{100}}{\textbf{10} + \textbf{2}} = \frac{\textbf{ml}_{\text{Titrant}}}{\textbf{120}}$$

HARDNESS IN FIRST-CARB AND THIN JUICE

Sampling interval: 4 times per shift

Hardness (limesalts) is the total of water-soluble salts of calcium (Ca) and magnesium (Mg). A high hardness content in thin juice (purified juice) causes a rapid formation of scale in evaporators. Therefore, the first-carb and thin juice should be tested for hardness content to evaluate the purification process with regard to hardness removal during purification. If the factory also has juice softening and molasses-desugaring processes, you should further test the soft thin juice and molasses for hardness content. To test a sample for hardness content, use the following procedure:

1. Filter the first-carb juice. Thin and soft thin juice does not need to be filtered, unless the sample is cloudy.

2. Cool the sample, apply the sample on the refractometer, and read the % *RDS*.
3. Pipet 10 ml of the sample to a porcelain dish, and add about 10 ml of hot DI water to it to reduce the intensity of the juice color.
4. Add 2 ml of **Reagent 8** (hardness buffer solution) and 2 to 3 shakes of **Reagent 9** (hardness indicator) to the dish to get a good **pink** color.
5. Stir well to dissolve the powder.
6. Titrate with **Reagent 10** (titrant, EDTA solution) to the first color change and stop. Proceed slowly (drop by drop) to the pure **blue** endpoint (see the following Note). 1 ml of titrant (0.036 EDTA) equals 1 mg CaO.

Calculate the hardness content according to the following formula:

$$\text{Hardness} = \frac{\text{ml}_{\text{Titrant}}}{\%\ RDS} \times 1000\,\text{mg CaO}/RDS$$

The preceding equation is based on the following calculation, considering the fact that each ml of **titrant** (EDTA solution) is equivalent to 1 mg or 0.001 g CaO:

$$\text{Hardness} = \text{ml}_{\text{Titrant}} \times 0.001 \times \frac{100}{\text{ml}_{\text{Sample}}}\,\text{g}/100\,\text{ml}$$

For 10 ml of sample,

$$\text{Hardness} = \text{ml}_{\text{Titrant}} \times 0.001 \times \frac{100}{10} = \text{ml}_{\text{Titrant}} \times 0.01\,\text{g}/100\,\text{ml}$$

To find out the amount of hardness in 100 g dry substance (called g hardness per *RDS*), apply the *RDS* and the density of the sample:

$$\text{Hardness} = \frac{\text{ml}_{\text{Titrant}} \times 0.01}{\frac{\%\ RDS \times d}{100}} = \frac{\text{ml}_{\text{Titrant}}}{\%\ \text{RDS} \times d}\,\text{g CaO}/RDS$$

or

$$\text{Hardness} = \frac{\text{ml}_{\text{Titrant}}}{\%\ RDS \times d} \times 1000\,\text{mg CaO}/RDS$$

The density can be omitted if its value is close to 1 (e.g., in first- and second-carb juice). Therefore, the equation becomes

$$\text{Hardness} = \frac{\text{ml}_{\text{Titrant}}}{\%\ RDS} \times 1000\,\text{mg CaO}/RDS$$

Therefore, we can recalculate the result in mg CaO in 100 ml of sample. For example, if hardness equates to 250 mg/*RDS*, and *RDS* to 15.5, the mg hardness in 100 ml juice is

$$(\text{mg CaO}/RDS) \times \frac{RDS}{100} = 250 \times \frac{15.5}{100} = 38.5\,\text{mg CaO}/100\,\text{ml}$$

or 385 ppm (385 mg CaO/1000 ml).

NOTES

- **If the endpoint is poor, try the following remedies:**
 - **Use hot distilled water in step 3 because endpoint sharpens and is easier to see**
 - **Stir well during titration (step 6) to precipitate all of Mg^{2+} as $Mg(OH)_2$**
 - **Add 4 ml of Reagent 8 instead of 2 ml**
 - **Keep the buffer tightly capped to avoid loss of its volatile components.**
- **If the endpoint fades, there is a slow drifting back from blue to pink. This can happen for the following reasons:**
 - **You have too much buffer. Correct this by using less buffer.**
 - **The amount of $Mg(OH)_2$ precipitate is insufficient. Correct this by using hot water for makeup.**
 - **If the problem still exists after trying the first and second reasons corrective measures, it is possible that the endpoint is not yet reached; that is, the hardness indicator is not stable under alkaline conditions. Therefore, the titration should be continued until the color does not change back to pink.**
- **Drop titration: If the hardness is very low (when factory has the softening process), you must titrate the sample by drops with the help of a dropper instead of a buret. In this case, the formula should be divided by 20 because *each ml equals almost 20 drops*:**

$$\text{Hardness} = \frac{\text{ml}_{\text{Titrant}}}{\%\ RDS} \times \frac{1000}{20} = \frac{\text{Drop}_{\text{Titrant}}}{\%\ RDS} \times 50 \quad \text{mg Cao}/RDS$$

CARBONATION-LIME RESIDUE TESTS

Sampling interval: Once from each filter per shift

Carbonation-lime residue (limecake) is the precipitate of the first-carb operations. The amount of sugar left in the lime reside is important because it is one of the major sugar losses in the factory. Carbonation-lime residue is analyzed for the following tests:

- Sugar content
- CaO content
- Moisture content

To test **carbonation-lime residue** for **sugar content**, proceed as follows

1. Weigh 52.0 g of the sample in a plastic 400-ml beaker.
2. Add 177 ml of **Reagent 11** (2.5% aluminum chloride solution).
3. Mix it well.
4. Let it set for about 3 minutes and mix it again.
5. Filter it into a clean beaker and return the first unclear portion (about the first 10 ml) of the filtrate back to the funnel.
6. Rinse a polarimeter tube with clear filtrate, fill the tube, and read the polarimetric reading when the display has stabilized.

When a 100-mm polarimeter tube is used, the result is

$$\%\ \text{Sugar} = \text{Polarimetric reading} \times 2$$

Carbonation-lime residue (limecake) discharged from press filters contains about 80% CaO. This test is important for calculating the material balance of the factory. To test **carbonation-lime residue** for **CaO content**, proceed as follows:

1. Weigh 5.0 g of sample in a weighing dish.
2. Transfer the sample to a porcelain dish by washing with DI water using a plastic wash bottle.
3. Add 25 ml of **Reagent 12** (1.78 *N* nitric acid).
4. Boil for 3 to 4 minutes to expel all CO_2 gas from the decomposition of carbonates.
5. Cool to the standard temperature, add 2 to 3 drops of **Reagent 5** (phenolphthalein solution), and titrate with **Reagent 13** (1.78 *N* sodium hydroxide solution) to a **pink** color (neutrality equal to pH = 7).

Calculate % CaO according to the following equation:

$$\%\ \mathrm{CaO} = \frac{(25 - \mathrm{ml}_{\mathrm{Hydroxide}}) \times 0.05}{\mathrm{Mass}_{\mathrm{Sample}}} \times 100 = \frac{(25 - \mathrm{ml}_{\mathrm{Hydroxide}}) \times 0.05}{5.0} \times 100 = 25 - \mathrm{ml}_{\mathrm{Hydroxide}}$$

Carbonation lime (limecake) discharged from cake filters contains about 50 to 70% water, depending what type of filter is used. To test **carbonation lime** for **moisture content** proceed as follows:

1. Weigh a stainless steel moisture dish with its cover (W_1).
2. Add about 10.0 g of sample into the weighed dish and weigh the dish plus the cake (W_2).
3. Place the dish and cover (remove the cover) in a drying oven at 105°C for 1 hour.
4. Replace the cover on the dish, remove the dish and cover from the oven, and place it in the desicator for 10 minutes to cool down, and then weigh (W_3).

Calculate % moisture according to the following equation:

$$\%\ \mathrm{Moisture} = \frac{W_2 - W_3}{W_2 - W_1} \times 100$$

NOTE

Moisture analyzers can be used for the limecake moisture test to obtain a faster result than by the oven method. Moisture analyzers are equipped with a heating element and a microbalance. The analyzer automatically weighs the sample before and after drying and calculates the percentage of moisture. Today, many sugar labs use moisture analyzers to determine the moisture in carbonation-lime residue (limecake) and pulp samples. For the detailed procedure, see the Moisture in Pulp section.

CaO IN DIFFERENT JUICES

(Note: This procedure can be applied on mixed, pre-carb, and second-carb juice and on the returned mud sample.)

The percentage of CaO in juice indicates the amount of lime (as CaO) used in different stages of purification. The determination of CaO is based on the reaction of acid with lime, and the neutralization of excess acid by an alkaline solution. To test a sample for CaO content, use the following procedure:

1. Pipet 10 ml of unfiltered juice to a porcelain dish.
2. Add a measured amount of **Reagent 14** (0.36 *N* sulfuric acid) from a buret. For example, to mixed juice add 15 ml of acid.
3. If foam forms (as a result of CO_2 development), add a few drops of ether.
4. Add a few drops of **Reagent 7** (special indicator) and titrate with **Reagent 15** (0.36 *N* sodium hydroxide) to the **green** endpoint.

Calculate the % CaO according to the following equation:

$$\%\ \mathrm{CaO} = (\mathrm{ml}_{\mathrm{Acid}} - \mathrm{ml}_{\mathrm{Hydroxide}}) \times 0.01 \times \frac{100}{\mathrm{ml}_{\mathrm{Sample}}}$$

$$\%\ \mathrm{CaO} = (\mathrm{ml}_{\mathrm{Acid}} - \mathrm{ml}_{\mathrm{Hydroxide}}) \times 0.01 \times \frac{100}{10}$$

$$\%\ \mathrm{CaO} = (\mathrm{ml}_{\mathrm{Acid}} - \mathrm{ml}_{\mathrm{Hydroxide}}) \times 0.1$$

EXAMPLE 3

	Mixed Juice	First-Carb
Sample	10.0 ml	10.0 ml
Acid used	15.0 ml	25.0 ml
Hydroxide used	8.0 ml	7.0 ml

$(\mathrm{ml}_{\mathrm{Acid}} - \mathrm{ml}_{\mathrm{Hydroxide}}) \times 0.1 = (15.0 - 8.0) \times 0.1 = 0.7\%$ CaO in mixed juice

$(\mathrm{ml}_{\mathrm{Acid}} - \mathrm{ml}_{\mathrm{Hydroxide}}) \times 0.1 = (25.0 - 7.0) \times 0.1 = 1.8\%$ CaO in first-carb juice

You can calculate the amount of fresh lime (lime consumption) if the lime in the mixed-juice tank consists of return and fresh lime as follows:

1.8 – 0.7 = 1.1% CaO on juice brought in by milk of lime (fresh lime)

MOISTURE IN PULP

To test the amount of **moisture in pulp**, use the following procedure:

1. Weigh a stainless steel moisture dish with its cover (W_1).
2. Add about 10.0 g of sample into the weighed dish and weigh the dish plus cake (W_2).
3. Place the dish and cover (with the cover removed) in a drying oven at 105°C for 1 hour.
4. Replace the cover on the dish, remove the dish and cover from the oven, and weigh it hot (W_3). This step should be performed as rapidly as possible to prevent absorption of moisture by the hot dry sample.

Calculate the % moisture according to the following equation:

$$\%\ \mathrm{Moisture} = \frac{W_2 - W_3}{W_2 - W_1} \times 100$$

NOTE

Moisture analyzers can be used for pulp moisture analysis, giving faster and more error-free results than the oven method. Moisture analyzers can store several drying programs, so they can be programmed to run the moisture content in pulps (wet, pressed, and dry pulps). The moisture analyzers are based on different technologies such as **microwave**, which utilizes an infrared heating system. Another type is the **halogen analyzer**, which uses a special heating technique with even faster drying time than the microwave type. Today, many sugar labs use moisture analyzers to determine the moisture in carbonation-lime residue (limecake) and pulp samples.

THIN-JUICE TESTS

Sampling interval: 2 times per shift

Thin juice is the product of the purification station and is analyzed for the following tests:

- *RDS*
- Sugar content
- pH
- Color

To test a **thin-juice** sample for ***RDS*** (refractometric dry substance or Brix), use the following procedure:

1. Cool the sample to the standard temperature (20°C).
2. Apply the sample on the refractometer.
3. Read it directly for the % *RDS*.

To test a **thin-juice** sample for **sugar content**, use the following procedure:

1. Weigh 26.00 g of cold sample in a weighing dish.
2. Transfer into a 200-ml Kohlrausch flask by washing with DI water using a wash bottle.
3. Mix well by swirling.
4. If it is not cold, cool to the standard temperature (20°C).
5. Add DI water from a wash bottle to below the neck of the flask.
6. If foam is on the surface, add 2 drops of ether to clear the foam.
7. Raise the volume to the mark on the flask by applying the DI water with a dropper. Pay attention that the bottom of the meniscus and the line correspond exactly.
8. Stopper the flask and mix it well by inversion and shaking, allowing bubbles to escape from the flask.
9. Rinse a polarimeter tube twice with the sample and fill the tube again.
10. Set the polarimeter to 0, place the tube in the polarimeter, and record the reading when the polarimeter has stabilized.

When the 100-mm polarimeter tube is used, the result is

$$\% \text{ Sugar} = \text{Polarimetric reading} \times 4$$

Calculate the **purity** (*P*) using the purity formula:

$$\%\ P = \frac{\%\ \text{Sugar}}{\%\ RDS} \times 100$$

To test a **thin-juice** sample for **pH**, use the following procedure:

1. Cool the sample to the standard temperature (20°C).
2. Rinse the electrode of the pH meter and beaker with a portion of the cold sample.
3. Take a sufficient sample in the beaker and immerse the electrode in the sample.
4. Read the pH when the reading has stabilized.

To test a thin-juice sample for **color**, use the following procedure:

1. Filter the sample using a filter paper capable of removing particles larger than 20-µm (Whatman #4 filter paper or the equivalent).
2. Measure the *RDS* of the sample on the refractometer.
3. Fill a color tube (10 or 25.4 mm in diameter) with the filtered sample.
4. Fill another tube with **DI water** as the blank (reference).
5. Adjust the spectrophotometer reading to 0 at 420 nm with the blank using the *T* (transmittance) or *A* (absorbance) mode of the instrument.
6. Read the *T* or *A* of the sample.

Calculate the color expressed as ICUMSA 420 (**IU color**) as follows:

$$C_{\text{ICUMSA 420}} = 10^6 \frac{A}{b \times RDS \times d}$$

If you are using T mode,

$$C_{\text{ICUMSA 420}} = 10^6 \frac{2 - \log T}{b \times RDS \times d}$$

b Cell path length (sample tube diameter), in mm
d Density, from Table A.1 in the Appendix or from *RDS*-density regression equation ($d = 0.0055 \times RDS + 0.9714$)

EXAMPLE 4

Given:

Cell path-length (*b*, sample cell diameter)	25.4 mm
RDS	14.5
Transmittance (*T*)	17.2
Density (*d*)	

$d = 0.0055 \times RDS + 0.9714 = 0.0055 \times 14.5 + 0.9714 = 1.05$

Calculate:
Color of the sample

$$C = 10^6 \frac{2 - \log T}{b \times RDS \times d} = 10^6 \frac{2 - \log 17.2}{25.4 \times 14.5 \times 1.05} = 1977$$

NOTE

For additional explanation of the RDS-density equation and a fast way of calculating the color by a computer, refer to the Spectrophotometer subsection in Section 2 of this chapter.

THICK-JUICE TESTS

Sampling interval: 2 times a shift

Thick juice is the product of the evaporation station. It is usually analyzed for the following tests:

- *RDS*
- pH
- Color

To test a **thick-juice** sample for *RDS* (refractometer dry substance), use the following procedure:

1. Cool the sample to the standard temperature (20°C).
2. Apply the sample on the refractometer.
3. Read it for the % *RDS*.

NOTE

There is no need to test the thick juice for sugar in order to calculate its purity. The purity of thin juice does not change considerably when it is evaporated to make the thick juice. In other words, there is no considerable amount of nonsucrose formation or sugar decomposition during the evaporation process; therefore, the purity of the thick juice stays almost the same as the purity of the thin juice.

To test a **thick-juice** sample for **pH**, use the following procedure:

1. Cool the sample to the standard temperature.
2. Rinse the pH meter electrode and beaker with a portion of the cold sample.
3. Put sufficient amount of the sample in the beaker and immerse the electrode in the sample.
4. Read the pH when the reading has stabilized.

To test a **thick-juice** sample for **color**, use the following procedure:

1. Prepare a 100 ml solution of about 5% *RDS* by using the following procedure.

$$\text{Mass}_{\text{Syrup}} = \frac{RDS_{\text{Required}}}{RDS_{\text{Syrup}}} \times \text{Mass}_{\text{Required}}$$

$$\text{Mass}_{\text{Water}} = \text{Mass}_{\text{Required}} - \text{Mass}_{\text{Syrup}}$$

For example, if the *RDS* of thick juice is 52.5%, use the following to make 100 g of a 10% *RDS* solution:

$$\text{Mass}_{\text{Syrup}} = \frac{5.0}{52.5} \times 100 = 9.5\text{g of juice}$$

$$\text{Mass}_{\text{Water}} = 100 - 9.5 = 90.5\,\text{g of water}$$

This means that 9.5 g of the juice has to be mixed with 90.5 g DI water.

2. Filter the sample using a filter paper capable of removing particles larger than 20-μm (Whatman #4 filter paper or the equivalent).
3. Measure the *RDS* on the refractometer.
4. Fill a color tube (10 or 25.4 mm in diameter) with the filtered sample.
5. Fill another tube with DI water as the blank (reference).
6. Adjust the spectrophotometer reading to 0 at 420 nm with the blank by using the *T* (transmittance) or *A* (absorbance) mode of the instrument.
7. Read the *T* or *A* of the sample.

Calculate the color expressed as ICUMSA 420 (**IU color**) as follows:

$$C = 10^6 \frac{A}{b \times RDS \times d}$$

In using *T* mode, calculate

$$C = 10^6 \frac{2 - \log T}{b \times RDS \times d}$$

b Cell path length (sample tube diameter), in mm
d Density, from Table A.1 in the Appendix or from *RDS*-density regression equation ($d = 0.0055 \times RDS + 0.9714$)

EXAMPLE 5

Given:

b	25.4
RDS	4.9
T	38.2

Calculate:
Color of the sample

$$d = 0.0055 \times RDS + 0.9714 = 0.0055 \times 4.9 + 0.9714 = 1.0$$

$$C = 10^6 \frac{2 - \log T}{b \times RDS \times d} = 10^6 \frac{2 - \log 38.2}{25.4 \times 4.9 \times 1.0} = 3358$$

SUGAR TRACES IN BOILER FEEDWATER

The **sugar traces** test is important because it determines the presence of sugar in the boiler feedwater and condenser water. This test is quick to perform and ideally suited for a routine check. To test a water sample for the presence of sugar, use the following procedure:

1. If the sample is hot, cool it in a water bath to the standard temperature (20°C).
2. Fill a test tube (usually 16 mm in diameter and 125 mm long) with about 3 ml of the sample.
3. Add 5 drops of **Reagent 16** (alpha-naphtol solution) and mix well.
4. Slowly add 2 ml of **Reagent 17** (concentrated sulfuric acid) into the tube, holding the tube in an inclined position, **away from you**, so that the acid will run down the side to the bottom and form a separate layer. Acid is heavier, so it stays at the bottom.
5. Shake to mix, and stand at the standard temperature for about 1 minute.
6. If color does not appear immediately, roll the tube gently for few seconds in an upright position between the palms of your hands (**color intensifies with heating**).
7. Check again for a color change. No color indicates the absence of sugar. A **purple ring** at the intersection of the two layers indicates the presence of sugar. Report as follows:

 A light ring of purple color = **light**

 A distinct, but not heavy ring of purple color = **medium**

 A heavy ring of purple color = **heavy**

NOTE

The presence of sugar in boiler feedwater can damage the boiler's operation. Therefore, the person running this test should immediately let his or her supervisor know when there is a sign of sugar presence in the boiler feedwater.

CHAPTER
11

SECTION 5

SUGAR-END METHODS OF ANALYSIS

SUBSECTIONS

- Standard-Liquor Tests
- Massecuite Tests
- Syrup Tests
- High-Raw and Low-Raw Sugar Tests
- Molasses Tests

STANDARD-LIQUOR TESTS

Standard liquor is the product of melting high-raw sugar and low-raw sugar in thick juice to produce feed for the first-stage of crystallization (white crystallization). It is analyzed for the following tests:

- *RDS*
- Sugar content
- pH
- Color

To test a **standard-liquor** sample for ***RDS*** (refractometric dry substance or Brix), use the following procedure:

1. Cool the sample to the standard temperature (20°C).
2. Apply the sample on the refractometer.
3. Read the % *RDS*.

To test a **standard-liquor** sample for **sugar content**, use the following procedure:

1. Weigh 26.00 g of cold sample in a weighing dish.
2. Transfer the sample into a 200-ml Kohlrausch flask by washing with DI water using a wash bottle.
3. Mix well by swirling.
4. Cool to the standard temperature.
5. Add DI water from a wash bottle to below the neck of the flask.
6. If foam is on the surface, add 2 drops of ether to clear the foam.

Beet-Sugar Handbook, by Mosen Asadi
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Day: Date:

White pan								High-raw continuous pan								Hi-raw sugar		L-raw sugar
Massecuite				Hi-green		Hi-wash		Massecuite						Low-green				
Pan #	RDS	Purity	pH	RDS	Purity	RDS	Color	RDS 1	RDS 2	RDS 3	RDS 4	Purity	pH	RDS	Purity	Sugar	Color	Sugar
								Low Raw Continuous Pan										
								Massecuite						Molasses produced				
								RDS 1	RDS 2	RDS 3	RDS 4	Purity	pH	RDS	Sugar	Purity	pH	Color
								Hi-Raw Seed Magma				Standard Liquor				Granulated Sugar		
								Pan #	RDS	Purity	Time	RDS	Purity	pH	Color	Color	Screen	Moist
											7AM							
											8							
											9							
											10							
											11							
											12PM							
											1							
											2							
											3							
								Low-raw seed pans			4							
								Pan #	RDS	Purity	5							
											6							
											7							
											8							
											9							
											10							
											11							
											12AM							
											1							
								Stored extract			2							
								RDS	Purity	pH	3							
											4							
											5							
											6							

Figure 11.19 Sugar-end lab sheet

7. Raise the volume exactly to the mark on the flask by applying the DI water with a dropper.
8. Stopper the flask and mix it well by inverting and shaking, allowing bubbles to escape from the flask.
9. Rinse a polarimeter tube twice with the sample and fill the tube again.
10. Set the polarimeter to 0, place the tube in the polarimeter, and record the reading when the polarimeter has stabilized.

When a 100-mm polarimeter tube is used, the result is

$$\% \text{ Sugar} = \text{Polarimetric reading} \times 4$$

Calculate the **purity** (P) according to the purity equation:

$$\%\ P = \frac{\%\ \text{Sugar}}{\%\ RDS} \times 100$$

To test a **standard liquor** sample for **pH**, use the following procedure:

1. Rinse the pH-meter electrode and beaker with a portion of the cold sample (20°C).
2. Put sufficient sample in the beaker and immerse the electrode in the sample.
3. Read the pH when the reading has stabilized.

To test a **standard-liquor** sample for **color**, use the following procedure:

1. Prepare a 100 ml solution of about 5% *RDS* by using the following procedure:

$$\text{Mass}_{\text{Syrup}} = \frac{RDS_{\text{Required}}}{RDS_{\text{Syrup}}} \times \text{Mass}_{\text{Required}}$$

$$\text{Mass}_{\text{Water}} = \text{Mass}_{\text{Required}} - \text{Mass}_{\text{Syrup}}$$

 For example, if the *RDS* of standard liquor is 62.5%, use the following equation to make 100 g of a 5.0% *RDS*:

$$\text{Mass}_{\text{Juice}} = \frac{5.0}{62.5} \times 100 = 8\,\text{g}$$

$$\text{Mass}_{\text{Water}} = 100 - 8 = 92\,\text{g}$$

 This means that 8 g syrup has to be mixed with 92 g water.
2. Filter the sample using a filter paper capable of removing particles larger than 20-μm (Whatman #4 filter paper or the equivalent).
3. Measure the *RDS* of the prepared sample on the refractometer.
4. Fill a color tube (10 or 25.4 mm in diameter) with the filtered sample.
5. Fill another tube with DI water as the blank (reference).
6. Adjust the spectrophotometer reading to 0 at 420 nm with blank using the T (transmittance) or A (absorbance) mode of the instrument.
7. Read the T or A of the sample.

Calculate the color expressed as ICUMSA 420 (**IU color**) as follows:

$$C = 10^6 \frac{A}{b \times RDS \times d}$$

If you are using the T mode,

$$C = 10^6 \frac{2 - \log T}{b \times RDS \times d}$$

b	Cell path length (sample tube diameter), in mm
d	Density, from Table A.1 or from *RDS*-density regression equation ($d = 0.0055 \times RDS + 0.9714$)

EXAMPLE 6

Given:

b	25.4
RDS	5.1
T	39.2

Calculate:
Color of the sample

$$d = 0.0055 \times RDS + 0.9714 = 0.0055 \times 5.1 + 0.9714 = 1.0$$

$$C = 10^6 \frac{2 - \log T}{b \times RDS \times d} = 10^6 \frac{2 - \log 39.2}{25.4 \times 5.1 \times 1.0} = 3140$$

MASSECUITE TESTS

Massecuites consist of sugar crystals surrounded by syrup. White (A) massecuite has the highest purity (almost the same as standard liquor, about 92%) and low-raw massecuite the lowest (about 75%). The massecuites are analyzed for the following tests:

- *RDS*
- Sugar content
- pH

To test a **massecuite** sample for ***RDS*** (refractometer dry substance), use the following procedure:

1. Double-dilute the sample by weighing 50.0 g of sample in a beaker, adding DI water to give a final mass of 100.0 g.
2. Stir the sample first with a glass rod and then with a magnetic stirrer until the sample is completely dissolved (use a retriever to take the magnet out of the beaker after stirring).
3. Cool the sample in a water bath to the standard temperature.
4. Apply the sample on the refractometer and record the % *RDS*:

$$\%\ RDS = \text{Reading} \times 2$$

5. Keep the rest of the sample for the sugar and pH test.

To test a **massecuite** sample for **sugar content**, use the following procedure:

1. Weigh 26.00 g of the cold sample left from the double-diluted solution (step 5 of the dry substance test) in a weighing dish.
2. Transfer into a 200-ml sugar flask by washing with DI water using a wash bottle.
3. For light samples (the white massecuite), there is no need to add a clarifying reagent and to filter the sample (step 11 is not needed). For dark massecuite (the high-raw and low-raw massecuite), add 5 ml of **Reagent 1** (20% aluminum chloride solution) and 5 ml of **Reagent 2** (20% ammonium hydroxide solution).
4. Mix well by swirling.

5. If it is not already, cool to the standard temperature.
6. Add DI water from a wash bottle to below the neck of the flask.
7. If foam is on the surface, add 2 drops of ether to clear the foam.
8. Raise the volume to the mark on the flask by applying the DI water with a dropper.
9. Stopper the flask and mix it well by inverting and shaking, allowing bubbles to escape from the flask.
10. Allow it to stand for about one minute.
11. Filter it into a clean beaker and return the first unclear portion (about the first 10 ml) of the filtrate back to the funnel. Use 18-cm Whatman #4 filter paper or equivalent for filtration.
12. Rinse a polarimeter tube twice with the clear sample and fill the tube again.
13. Set the polarimeter to 0, place the tube in the polarimeter, and record the reading when the polarimeter has stabilized.

When a 100-mm polarimeter tube is used, the % sugar is given as follows:

$$\% \text{ Sugar} = \text{Polarimetric reading} \times 8$$

Calculate the **purity** (*P*) using the purity formula:

$$\% P = \frac{\% \text{ Sugar}}{\% RDS} \times 100$$

To test a **massecuite** sample for **pH**, use the following procedure:

1. Rinse the pH meter electrode and beaker with a portion of the sample left from the double-diluted solution (step 3 of the dry-substance test).
2. Put sufficient cold sample in the beaker and immerse the electrode in the sample.
3. Read the pH when the reading has stabilized.

SYRUP TESTS

High-green (A) and **high-wash syrups** are mother liquors from centrifuging the white (A) massecuite. Low-green (B) syrup is the mother liquor from centrifuging high-raw (B) massecuite. These syrups are analyzed for the following tests:

- *RDS*
- Sugar
- pH

To test a **syrup sample** for ***RDS*** (refractometer dry substance) content, use the following procedure:

1. Double-dilute the sample by weighing 50.0 g of the sample in a beaker and adding DI water to give a final mass of 100.0 g.
2. Stir the sample with a glass rod until it is completely mixed.

3. Cool the sample in a water bath to the standard temperature.
4. Apply the sample on the refractometer and record the reading.

$$\%\ RDS = \text{Reading} \times 2$$

5. Keep the rest of the sample for the sugar and pH tests.

To test a syrup sample for **sugar content**, use the following procedure:

1. Weigh 26.00 g of the sample left from the double-diluted solution (step 5 of the dry substance test) into a weighing dish.
2. Transfer into a 200-ml sugar flask by washing with DI water using a wash bottle.
3. For light samples (the high-wash, and high-green syrups), there is no need to add a clarifying reagent and to filter the sample (step 11 is not needed). For dark samples (the low-green syrup), add 5 ml of **Reagent 1** (20% aluminum chloride solution) and 5 ml of **Reagent 2** (20% ammonium hydroxide solution).
4. Mix well by swirling.
5. If the sample is not cold already, cool it to the standard temperature.
6. Add DI water from a wash bottle to below the neck of the flask.
7. If foam is on the surface, add 2 drops of ether to clear the foam.
8. Raise the volume to the mark on the flask by applying the DI water with a dropper.
9. Stopper the flask and mix it well by inverting and shaking, allowing bubbles to escape from the flask.
10. Allow it to stand for about one minute.
11. Filter into a beaker and discard the first unclear portion (about the first 10 ml) of the filtrate.
12. Rinse a polarimeter tube twice with clear sample and fill the tube again.
13. Set the polarimeter to 0, place the tube in the polarimeter, and record the reading when the polarimeter has stabilized.

When a 100-mm polarimeter tube is used, the sugar is

$$\%\ \text{Sugar} = \text{Polarimetric reading} \times 8$$

Calculate the **purity** (*P*) using the purity formula:

$$\%\ P = \frac{\%\ \text{Sugar}}{\%\ RDS} \times 100$$

Use the solution in step 3 of the sugar test and follow the general instructions (used in massecuite analyses) for the **pH** measurement.

HIGH-RAW AND LOW-RAW SUGAR TESTS

High-raw (B) sugar is the product from centrifuging high-raw (B) massecuite. Low-raw (**C**) sugar is the product from centrifuging low-raw (**C**) massecuite. These two sugars are then melted in thick juice to produce the standard liquor. The high-raw and low-raw sugars are analyzed for sucrose content.

To test a **high-raw** or **low-raw sugar** sample for **sugar content**, use the following procedure:

1. Weigh 26.0 g of the sample in a weighing dish.
2. Transfer into a 200-ml sugar flask by washing with DI water using a wash bottle.
3. Dissolve at a low speed using a platform shaker until it is completely dissolved.
4. Wash down the neck of the flask with DI water using a wash bottle.
5. For the high-raw sample, add 0.5 ml of **Reagent 1** (20% aluminum chloride solution) and 0.5 ml of **Reagent 2** (20% ammonium hydroxide solution). For the low-raw sample, add 1 ml of each reagent.
6. Mix well by swirling.
7. If the sample is not cold already, cool it to the standard temperature (20°C).
8. Add DI water from a wash bottle to below the neck of the flask.
9. If foam is on the surface, add 2 drops of ether to clear the foam.
10. Raise the volume to the mark on the flask by applying the DI water with a dropper.
11. Stopper the flask and mix it well by inverting and shaking, allowing bubbles to escape from the flask.
12. Allow it to stand for about one minute.
13. Filter into a beaker and return the first unclear portion (about the first 10 ml) of the filtrate back to the funnel. Use 18-cm Whatman #4 filter paper or the equivalent for filtration.
14. Rinse a polarimeter tube twice with the clear sample and fill the tube again.
15. Set the polarimeter to 0, place the tube in the polarimeter, and record the reading when the polarimeter has stabilized.

When a 100-mm polarimeter tube is used, the % sugar is

$$\% \text{ Sugar} = \text{Polarimetric reading} \times 4$$

MOLASSES TESTS

Molasses is the mother liquor from centrifuging low-raw massecuite. It is usually analyzed for the following tests:

- *RDS*
- Sugar
- pH
- Color (in the case that factory is equipped with the molasses-desugaring process)
- Hardness (in the case that factory is equipped with the molasses-desugaring process)

To test a **molasses** sample for ***RDS*** (refractometer dry substance) content, use the following procedure:

1. Double-dilute the sample by weighing 50.0 g of sample in a beaker and adding DI water to give a final mass of 100.0 g.
2. Stir the sample first with a glass rod and then with a magnetic stirrer until the sample is completely dissolved (use a retriever to take the magnet out of the flask).
3. Cool the sample in a water bath to the standard temperature.

4. Apply the sample on the refractometer and record the reading:

$$\%\ RDS = \text{Reading} \times 2$$

5. Keep the rest of the sample for the sugar, pH, and limesalts tests.

To test a **molasses** sample for **sugar content**, use the following procedure:

1. Weigh 26.00 g of the sample left from the double-diluted solution (step 5 of the dry-substance test) into a weighing dish.
2. Transfer into a 200-ml sugar flask by washing with DI water using a plastic wash bottle.
3. Add 5 ml of **Reagent 1** (20% aluminum chloride solution) and 5 ml of **Reagent 2** (20% ammonium hydroxide solution), and mix it well.
4. If the sample is not cold already, cool it to the standard temperature.
5. Add DI water to the neck of the flask using a wash bottle.
6. If foam is on the surface, add 2 drops of ether to clear the foam.
7. Raise the volume to the mark on the flask using a dropper.
8. Stopper the flask and mix it well by inverting and shaking, allowing bubbles to escape from the flask.
9. Let it set for about one minute.
10. Filter into a beaker and return the first unclear portion (about the first 10 ml) of the filtrate back to the funnel. Use 18-cm Whatman #4 filter paper or the equivalent for filtration.
11. Rinse a polarimeter tube twice with the clear filtrate and fill the tube again.
12. Set the polarimeter to 0, place the tube in the polarimeter, and record the reading when the polarimeter has stabilized.

When using a 100-mm polarimeter tube, the % sugar is

$$\%\ \text{Sugar} = \text{Polarimetric reading} \times 8$$

Calculate the purity according to the purity equation:

$$\%\ P = \frac{\%\ \text{Sugar}}{\%\ RDS} \times 100$$

To test a **molasses** sample for **pH**, use the following procedure:

1. Rinse the pH meter electrode and beaker with a portion of the sample left from double-diluted solution (step 3 of the dry-substance test).
2. Put sufficiently cold sample in the beaker and immerse the electrode in the sample.
3. Read the pH when the reading has stabilized.

To test a **molasses** sample for **color**, use the following procedure:

1. Prepare 100 ml solution of about 2% *RDS* by using the following procedure.

$$\text{Mass}_{\text{Molasses}} = \frac{RDS_{\text{Required}}}{RDS_{\text{Molasses}}} \times \text{Mass}_{\text{Required}}$$

$$\text{Mass}_{\text{Water}} = \text{Mass}_{\text{Required}} - \text{Mass}_{\text{Molasses}}$$

For example, if the *RDS* of molasses is 80.5, you can make 100 g of a 2% *RDS* solution as follows:

$$\text{Mass}_{\text{Molasses}} = \frac{2.0}{80.5} \times 100 = 2.5\,\text{g}$$

$$\text{Mass}_{\text{Water}} = 100 - 2.5 = 97.5\,\text{g}$$

This means that 2.5 g molasses has to be mixed with 97.5 g water.

2. Filter the sample using a filter paper capable of removing particles larger than 20-μm (Whatman #4 filter paper or the equivalent).
3. Measure the % *RDS* of the prepared sample on the refractometer.
4. Keep part of the sample for the hardness test.
5. Fill a color tube (10 or 25.4 mm in diameter) with the filtered sample.
6. Fill another tube with DI water as the blank (reference).
7. Adjust the spectrophotometer reading to 0 at 420 nm with blank using the *T* (transmittance) or *A* (absorbance) mode of the instrument.
8. Read *T* or *A* of the sample.

Calculate the color expressed as ICUMSA 420 (**IU color**) as follows:

$$C = 10^6 \frac{A}{b \times RDS \times d}$$

If you are using the *T* mode, calculate the color value by the following equation:

$$C = 10^6 \frac{2 - \log T}{b \times RDS \times d}$$

b Cell path length (sample tube diameter), in mm

d Density, from Table A.1 in the Appendix or from *RDS*-density regression equation ($d = 0.0055 \times RDS + 0.9714$)

EXAMPLE 7

Given:

RDS	1.9%
b	25.4 mm
$d = 0.0055 \times RDS + 0.9714 = 0.0055 \times 1.9 + 0.9714 = 1.0$	
T	2.2

Calculate:

Color of the sample

$$d = 0.0055 \times RDS + 0.9714 = 0.0055 \times 1.9 + 0.9714 = 1.0$$

$$C = 10^6 \frac{2 - \log T}{b \times RDS \times d} = 10^6 \frac{2 - \log 2.2}{25.4 \times 1.9 \times 1.0} = 34347$$

To test a **molasses** sample for **hardness content**, use the following procedure:

1. To the sample left from the color test (step 4 in the color test), add 2 ml of **Reagent 8** (hardness buffer solution) and 2 to 3 shakes of **Reagent 9** (hardness indicator) to the dish to obtain a good **pink** color, and stir it well to dissolve the indicator. If the color solution is still dark, add about 10 ml more DI water to reduce the intensity of the color.
2. Titrate with **Reagent 10** (EDTA, hardness titrant) to the first color change and stop. Proceed slowly (drop by drop) to the pure **blue** endpoint. (Note: The endpoint should not fade.)

Calculate the **hardness** (limesalts) according to the following formula (for more information regarding the equations, refer to hardness determination in the Beet-end Analyses section).

$$\text{Hardness} = \frac{\text{ml}_{\text{Titrant}}}{RDS} \times 1000\,\text{mg CaO}/RDS$$



Figure 11.20 20 drops of water is about 1 ml

NOTES

- **Molasses hardness is usually expressed as *mEq/RDS* (milliequivalent per 100g *RDS* of the juice) and calculated by the following formula:**

$$\text{Equivalent mass} = \frac{\text{Molecular mass(CaO)}}{\text{Valence(CaO)}} = \frac{56}{2} = 28$$

$$\text{Hardness} = \frac{\text{ml}_{\text{Titrant}}}{RDS} \times \frac{1000}{28} = \frac{\text{ml}_{\text{Titrant}}}{RDS} \times 35.7\ mEq/RDS$$

- **Drop titration: If the hardness is very low, titrate the sample with a dropper instead of a buret. In this case, the formula is divided by 20 because each ml equals almost 20 drops (see Figure 11.20):**

$$\text{Hardness} = \frac{\text{Drop}_{\text{Titrant}}}{RDS} \times \frac{35.7}{20} = \frac{\text{Drops}_{\text{Titrant}}}{RDS} \times 1.8\ mEq/RDS$$

CHAPTER
11

SECTION *6*

QUALITY-CONTROL METHODS OF ANALYSIS

SUBSECTIONS

- Granulated-Refined Sugar Tests
- Powdered Sugar Tests

Quality-control tests refer to analyses of **granulated-refined** (GR) sugar and specialty sugars (e.g., powdered sugar, brown sugar, and cube sugar). The analytical tests are routine for GR sugar but not so for specialty sugars because a sugar factory may produce one, more, or none of the specialty sugars.

This section provides the analytical tests for controlling the quality of GR sugar, powdered sugar, and brown sugar. Sampling intervals of these products are not discussed because they differ from factory to factory.

GRANULATED-REFINED SUGAR TESTS

Granulated-refined sugar (GR sugar) is the product of centrifuging white massecuite. After centrifuging, the sugar goes to the sugar dryer to lose its moisture. Then the sugar goes to the sugar cooler to be conditioned for storage in the silo or for packing. After the cooling process, a GR sugar sample must be taken from a freely falling place. You should take the sample in the entire width because flowing sugar forms a heap, in which larger crystals move toward the outer lower part and finer particles remain in the center. Usually, GR sugar is tested for the following properties:

- Color
- Crystal size
- Moisture
- Ash
- Specks
- Sediment
- Floc
- Sulfur dioxide
- Microorganisms

Beet-Sugar Handbook, by Mosen Asadi
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Color of Sugar

To test a GR sugar sample for color, use the following procedure:

1. Weigh 50.0 g sugar in a weighing dish.
2. Pour the sugar into an Erlenmeyer flask.
3. Add 50.0 g DI water to the flask and mix with the help of a magnetic stirrer until the sugar is dissolved.
4. Filter the sample into a clean flask using a filter paper capable of removing particles larger than 20 µm (Whatman #4 filter paper or the equivalent). Discard the first unclear portion (about the first 10 ml) of the filtrate.
5. Measure the *RDS* of the sample on the refractometer.
6. Remove entrained air by letting the solution set for a few minutes.
7. Fill a clean and dry color tube (1.0 or 2.5 cm in diameter) with the sample.
8. Fill another matched tube with DI water as the blank (reference).
9. Adjust the spectrophotometer reading to 0 at 420 nm with the blank using the *T* (transmittance) or *A* (absorbance) mode of the instrument.
10. Read the *T* or *A* of the sample.

Calculate the color expressed as ICUMSA 420 as follows:

$$C = 10^6 \frac{A}{b \times RDS \times d}$$

If you are using the *T* mode,

$$C = 10^6 \frac{2 - \log T}{b \times RDS \times d}$$

b Cell path length (sample tube diameter), in mm
d Density, from Table A.1 in the Appendix or from *RDS*-density regression equation ($d = 0.0055 \times RDS + 0.9714$)

EXAMPLE 8

Given:

RDS	50.2%
T	88.8
b	25.4 mm

Calculate:
Color of sugar

The density can be obtained from the *RDS* value using Table A.1, as 1.23 or calculated from the *RDS*-density regression equation:

$$d = 0.0055 \times RDS + 0.9714 = 0.0055 \times 50.2 + 0.9714 = 1.25$$

$$C = 10^6 \frac{2 - \log T}{b \times RDS \times d} = 10^6 \frac{2 - \log 88.8}{25.4 \times 50.2 \times 1.25} = 32$$

NOTE

- **For more explanation on the RDS-density equation and a "fast" way of calculating color by computer, refer to the Spectrophotometer subsection in Section 2 of this chapter.**

Crystal Size of Sugar (One-Screen Method)

The crystal size test of sugar is carried out with one screen (sieve) for quick control of crystallization process. The screen aperture (opening) size depends on the size of the crystals produced by the individual sugar factory. In the United States, most factories use screen number 35 U. S. standard (equal to 32 Tayler mesh), which has an opening of 0.5 mm (500 microns). The word **mesh** denotes the number of openings per unit of surface. To test a sugar sample for crystal size (quick method), use the following procedure:

1. Weigh a clean and empty 35-screen and record its weight (M_1).
2. Place the screen on the bench-top sieve shaker (Ro-Tap sieve shaker).
3. Put the weighing dish on the balance, zero the balance, and weigh 100.0 g sugar.
4. Empty the sugar from the weighing dish on the screen.
5. Cover the screen with the top lid, set it on the shaker, and shake for 5 minutes.
6. Remove the screen from the shaker, reweigh it with the sugar, and record the mass (M_2).

Calculate the % sugar retained on the screen and passing through as follows:

$$\% \text{ On} = M_2 - M_1$$

$$\% \text{ Pass} = 100 - \% \text{ on}$$

EXAMPLE 9

$M_1 = 282.3$ g
$M_2 = 301.4$ g

$$\% \text{ On} = M_2 - M_1 = 296.9 - 282.1 = 14.8$$

$$\% \text{ Pass} = 100 - \% \text{ on} = 100 - 14.8 = 85.2$$

NOTES

- **The crystal size test is suitable for both GR sugar and raw sugar. In the testing of the raw sugar, the mother liquor should be washed with ethanol before the crystal size test.**
- **You may use a vacuum-type machine instead of the Ro-Tap sieve shaker.**

Crystal Size Distribution (Multiple-Screen Method)

The **crystal size distribution** (CSD) test with multiple screens is usually performed on a daily sugar sample composite using five screens. The size of the screens depends on the crystal size of the sugar produced by the individual factory. (See the following Note for choosing the screen size.) To test a GR sugar sample for CSD, use the following procedure:

1. Weigh the bottom pan and each of the screens and record their mass.
2. Stack the bottom pan and screens on the bench-top sieve shaker (Ro-Tap sieve shaker) in the correct size order (the largest opening at the top and the smallest at the bottom).
3. Put the weighing dish on the balance, zero the balance, and weigh 100.0 g sugar.
4. Pour the sugar from the weighing dish on the top screen.
5. Cover the screen with the top lid and shake it for 5 minutes.
6. Remove the screens from the shaker, reweigh them, and record the mass.
7. Calculate the percentage remaining on each screen by deducting the second weight (the weight of the screen and the sugar) from the first weight (the weight of empty screen).

The results of the screen test are used to calculate:

- Mean aperture
- Coefficient of variation

The **mean aperture** (*MA*) expresses the average crystal size of the sugar. *MA* is usually expressed as the screen size (in mm or micrometer) on which 50% of the sample stays, and 50% passes through.

The **coefficient of variation** (*CV*) expresses the **uniformity** (distribution) of crystals. In general, *CV* is a way of describing the precision of the result of a set of measurements. A low *CV* value characterizes a better uniformity. A *CV* of less than 35% is desirable for sugar crystals.

Cumulative percentages remaining on the screens are used to calculate the *MA* and *CV* as follows:

- Calculate the cumulative percentages remaining on each screen.
- Plot these values against the corresponding screen **aperture** (the opening size of the screen) on arithmetic probability paper.
- Draw a straight line through the points obtained.
- Read the size of the aperture corresponding to 50% and 16%:

$$MA = \text{Aperture corresponding to } 50\%$$

$$CV = \frac{\text{Aperture corresponding to } 16\% - MA}{MA} \times 100$$

NOTES

- **The total weight of the top two screens, after screening, should be 10 to 20% of the sample, and the total two bottom screens, 70 to 80% of the sample. If this is not the case, different screens should be used to obtain these values. The very top and very bottom screens should be chosen in a way that the sugar remaining on each does not exceed 1% of the sample. Therefore, the percentages of powder (called dust) and very coarse particles (lumps) can be determined. The amount of dust and lumps is important to customers, so care must be taken during the crystallization process to keep these fraction within the specifications.**
- **Subsampling is carried out in some factories to obtain more representative and homogeneous samples before crystal size testing. First, a gross sample of sugar is taken. Then the sample is reduced to a representative lab sample. For subsampling, a riffle sampler may be used before the crystal size**

test. The riffle sampler is a metal box about 40 × 30 × 40 cm divided by 24 compartments. The sample is poured to the top of the riffle through the compartments and cut into two drawers. The drawers are then removed and re-poured until the desired mixed and homogeneous sample is obtained.

- **The subsampling can also be achieved by coning. First, the sample is formed into a cone. Next, the sample is poured onto the top of the cone. The result is then mixed and flattened, and a new cone is formed. As each successive sample is added to the cone, the total is mixed thoroughly and a new cone is formed. After all the samples have been mixed by coning, the mass is flattened and a layer of sugar is formed.**
- **Cornering is an easier way of making the sample more homogeneous. In cornering, the sample is poured onto a paper sheet, then leveled, and mixed by pulling each corner over to its opposite corner. This procedure is repeated several times. To collect the sample, all four corners of the sheet should be simultaneously raised and the sample taken from the middle.**
- **Crystal size and crystal size distribution (CSD) expressed by the *MA* and *CV* equation is based on the assumption that sugar has a normal size distribution (the relation between cumulative percentages remaining on the screens and the corresponding aperture size of the screens is linear).**
- **The *MA-CV* method is suitable for both GR sugar and raw sugar when they have normal size distribution. It does not work for powdered sugar. Raw sugar should be washed with ethanol before the crystal size test to remove the mother liquor from the crystals. For this purpose, the raw sugar is soaked in 90% ethanol for 1 hour (with occasional shaking of the sample), and then the sample is filtered under a vacuum. (Note that the sucrose is not soluble in ethanol, so there is no concern regarding the change in size of the crystals.) It is usual to express the result of the crystal size test for beet raw sugar as a percentage through a screen with a 0.400 mm (400 μm; equal to 40 ASTM) opening and for cane raw sugar through a screen with a 0.600 mm (600 μm; equal to 30 ASTM) opening.**

Moisture in Sugar

The **moisture** content of granulated-refined (GR) sugar must not exceed 0.03%. To test a GR sugar sample for moisture content, proceed as follows:

1. Put a clean, dry stainless-steel moisture dish (with cover removed) in the oven for 30 minutes.
2. Remove from the oven and place the dish and its cover in a desicator to cool to room temperature.
3. Weigh the dish with its cover to the nearest 0.0001 g (W_1).
4. Add about 10.0 g of sample into the weighed dish and weigh the dish plus the sugar (W_2).
5. Place the dish and cover (remove the cover) in the drying oven at 105°C for 1 hour (or until constant weight is achieved).
6. Replace the cover on the dish, remove the dish and cover from the oven, place the covered dish in the desicator for 10 minutes to cool down, and then weigh to the nearest 0.0001 (W_3).

Calculate % moisture by the following equation:

$$\%\ \text{Moisture} = \frac{W_2 - W_3}{W_2 - W_1} \times 100$$

Ash in Sugar

Ash refers to water-soluble salts of inorganic and organic compounds. The ash content of a GR sugar sample is measured by a conductivity meter. Ash determined by conductivity is called **conductivity ash**. Conductivity of a solution of inorganic compounds is very high, but that of a pure sucrose solution in distilled, or DI, water is very low. Therefore, the conductivity of an impure sucrose solution is due almost entirely to the amount of ash. To test a sugar sample for ash content, use the following procedure:

1. Weigh 28.0 g of sugar in a weighing dish (see the Notes).
2. Place a beaker on the balance and zero it.
3. Pour the sugar into the flask and add DI water to bring up the weight to 100.0.
4. Insert a magnet and completely dissolve the sugar.
5. Put the DI water into the conductivity tube and let it cool to 20°C.
6. Read the conductivity of the DI water and register the number (C_1).
7. Pour out the distilled water from the tube and pour in the sugar solution.
8. Wait for the temperature to adjust to 20°C and read the conductivity (C_2).

Calculate % ash according to the following formula:

$$C = C_2 - 0.35C_1$$

$$\% \text{ Ash} = C \times 0.0006$$

NOTES

- **The standard concentration for the conductivity measurement is 28% m/m (this is equivalent to 31.3% m/v). At this concentration, the conductivity is at its maximum.**
- **Potassium chloride solution is used for checking the conductivity meter. For making 0.0002 mole/L of this solution, first make a 0.01 mole/L by weighing 745.5 mg of dehydrated potassium chloride (dehydrate it at a dull red heat on the gas burner) in 1 L. Dilute 10 ml of this solution in a 500-ml flask to make a 0.0002 mole/L. This solution has a conductivity of 26.6 μS/cm (microsiemens per centimeter) at 20°C (after deduction of the specific conductivity of the water used).**
- **If the determination cannot be made at the standard temperature of 20°C, the correction can be performed by the following formula:**

$$C_{20} = \frac{C_T}{1 + 0.026(T - 20)}$$

C_T Conductivity at temperature T

Specks in Sugar

Specks are materials of dark brown and black color in the sugar that are distinguishable by the naked eye. A piece of caramelized sugar is an example of a speck. Specks are determined by visual inspection by using a board (about 50 by 50 cm) framed about 5 cm high and painted gloss white. To test a sugar sample for the presence of specks, use the following procedure:

1. Place 300 g sugar on the board and spread the sample over the entire surface of the board.
2. Count any visible specks.

Calculate the counted specks in 100 grams:

$$\text{Visible specks per } 100\,\text{g} = \frac{\text{Visible specks counted}}{3}$$

NOTE

The specification for specks is 3 in 100 g sugar. Any speck rating in excess of 3 requires immediate correction.

Sediment in Sugar

Sediment consists of water-insoluble and suspended substances in sugar with particles not distinguishable to the naked eye, for example, pan scale, rust particles, filter aid, and calcium deposits.

The filtration method is used to determine the amount of sediment. Usually, two filter papers (DISCS) are used: a **white filter** for observation of dark sediment and a **black filter** for lighter colored specs. In some sugar labs, half the filter paper is darkened with Indian ink. In this case, only one filter (half white and half black) is used for the sediment test. To test a sugar sample for sediment content, use the following procedure:

1. Weigh 300 g of sugar and put it in a 500-ml flask.
2. Add hot distilled water to the mark on the flask.
3. Stir until all the sugar has dissolved.
4. Moisten a filter paper with DI water and place it in the filter holder under a vacuum.
5. Filter the sugar solution.
6. Wash the filter with about 300 ml of hot DI water.
7. Compare the filter with the standard sediment discs (the procedure for making them follows) and mark the result as A-0 to A-4 (for white sediment) and B-0 to B-4 (for black sediment).

Specifications up to number 3 (0 contains the least and 4 the highest sediment) are acceptable, meaning A-0 through A-3 and B-0 through B-3. Any sediment (white or black) that matches the number 4 standard won't pass the sediment test. These are

A-1, B-4
A-2, B-4
A-3, B-4
A-4, B-1
A-4, B-2
A-4, B-3
A-4, B-4

The following reagents and equipment are needed for preparation of **standard sediment discs** (filter paper):

- Vacuum pan scale
- Fly ash
- Factory filter aid

- Sodium pyrophosphate ($Na_2P_2O_7$)
- Methyl cellulose solution: Stir 10 g in 1000 ml DI water for 2 hours. Let settle and filter it.

Use the following procedure for making the discs:

1. Weigh 0.09 g pan scale and 0.01 g fly ash. (These two sediments indicate themselves on the white side of the filter.)
2. Weigh 0.1 g filter aid. (This will show on the black side of the filter.)
3. Add the three previous materials to 1000 ml of DI water.
4. Add 2.5 g sodium pyrophosphate to the flask and let the magnetic stirrer stir continuously. This stock solution contains 100 ppm each of white and black sediments. (Note: Sodium pyrophosphate is not considered a part of sediment.)
5. Make the following dilutions (be sure the stock solution is well stirred during the dilutions):

5 ml of stock to 1000 ml	0.5 ppm
10 ml of stock to 1000 ml	1.0 ppm
15 ml of stock to 1000 ml	1.5 ppm
20 ml of stock to 1000 ml	2.0 ppm
30 ml of stock to 1000 ml	3.0 ppm
40 ml of stock to 1000 ml	4.0 ppm

6. Under a vacuum, filter each solution on a separate disc. As the liquid above the filter comes within 1 cm of the disc paper, quickly pipet 2 ml of the methyl cellulose solution into the remaining solution. Make sure that the sediment on the disc is not disturbed.
7. Dry the discs, attach them to a labeled paper with two-sided tape, and put them between two plates of glass. Prepare more discs than needed each time in case some discs have an uneven distribution of sediment.

Floc in Sugar

Floc refers mainly to **saponin**, which comes from the sugarbeet skin, passes all the processes, and ends up in sugar. If sugar contains more than 2 ppm saponin, it produces visible flocs after standing in an acidified solution for 1 to 8 days. Sugar with high floc content is not appealing to customers, particularly beverage producers. Flocs are divided in two types. One is visible in an acidified solution and the other is visible in alcoholic solutions. The following procedure is used to detect floc in an acidified solution:

1. Place 70 g of sugar into a quarter-liter glass canning jar.
2. Add 170 ml DI water and stir to dissolve the sugar.
3. Adjust the pH to 2.0 with diluted phosphoric acid.
4. Cap the jar loosely and place the jar in a boiling-water bath for 15 minutes.
5. Remove the jar from the bath, tighten cap, and set it aside for 24 hours. (Do not shake the jar during this period.)
6. After the prescribed time, look for the presence of floc. (Use a light to illuminate the sample for better observation).

NOTE

Floc measuring 2 ppm or less in sugar does not make any visual impression.

Sulfur Dioxide in Sugar

Sulfur dioxide (SO_2) is added to some of in-process products (juices, syrups, and liquors) mainly to reduce the formation of coloring materials. Therefore, a trace of sulfur might be present in the sugar, and this has to be determined because of food agency regulations (e.g., FDA regulations) regarding the maximum amount of sulfur in food products (see the following Note). The method described here is based on colorimetry. In this method, the reaction of sulfite and **rosaniline** in the presence of formaldehyde creates a color that is dependent on the amount of sulfur. You can measure the color by spectrophotometer and the amount of sulfur with the help of a standard graph. The following reagents are needed for the SO_2 test:

- **Rosaniline hydrochloride solution:** Suspend about 1 g Rosaniline (Basic Fuchsin) in distilled water, heat to about 50°C, and allow the solution to cool while shaking it. Then let it stand for 48 hours with occasional shaking. Next, filter and transfer 4 ml of the filtrate into a 100-ml flask. Add 1 ml of concentrated HCl and dilute to the mark on the flask. The solution can be used after 1 hour.
- **Formaldehyde solution 0.2%:** Dilute 20 ml of 10% formaldehyde to 1 L of DI water.
- **Sodium hydroxide solution 0.1 *N*:** Dissolve 4 g of NaOH and dilute to 1 L with DI water.

The following procedure explains the determination of SO_2 in a sugar sample:

1. Dissolve 25 g of sugar in a 100-ml flask to the mark with DI water.
2. Transfer 10 ml of this solution into a 10-mm test tube.
3. Take another tube and put in 10 ml DI water (blank)
4. To each tube, add the following solutions:
 0.2 ml NaOH solution
 2.0 ml rosaniline solution
 2.0 ml formaldehyde solution
5. Mix and allow the solutions to stand for about 30 minutes.
6. Set the spectrophotometer at 560 nm and absorbance mode.
7. Insert the blank into the spectrophotometer and set it to zero.
8. Remove the blank and insert the samples, measure the absorbance, and find the ppm SO_2 using a **standard graph** (the procedure for constructing such a graph follows). The results correspond to ppm SO_2 in sugar.

For a **standard graph**, prepare a series of sodium sulfate (Na_2SO_3) solution containing 2 to 20 ppm SO_2. First make a stock solution of 1000 ppm of SO_2 by dissolving 1.97 g Na_2SO_3 per liter DI water:

$$\frac{\text{Molecular mass}_{Na_2SO_3}}{\text{Molecular mass}_{SO_2}} = \frac{126}{64} = 1.97\,\text{g/L}$$

This solution contains 1 g SO_2 per liter (1000 mg/L) or 1000 ppm. For making 0, 1, 2, 4, 8, 10, and 20 ppm solutions, pipet 0, 1, 2, 4, 8, 10, and 20 ml, respectively, of stock solution into 1000-ml flasks, add 4 ml NaOH solution to each flask, and raise the solution to the mark with DI water. Pipet 10 ml of each calibration standard into a 10-mm test tube and add the following solutions:

- 1.0 ml rosaniline solution
- 1.0 ml formaldehyde solution

Let the solution stand at room temperature for about 30 minutes and measure the absorbance (A) at 560 nm against the blank (solution with zero ppm standard solution). Plot a graph of absorbance (A) against ppm, as shown in Figure 11.21. As you can see from the chart, the regression equation of this relationship is $Y = 9.2337 \times A - 0.3233$

NOTE

The FDA requires that the amount of sulfur dioxide in food products, including sugar, not exceed 20 ppm. Other standards, such as the Codex Alimentarius Standard, specify a maximum level of 15 ppm. Therefore, sugar should be tested for sulfur dioxide content in the laboratory routinely to adjust the amount of sulfur dioxide used during operations.

POWDERED SUGAR TESTS

Powdered sugar is produced by milling (grounding) granulated-refined sugar (directly after drying in the sugar dryer or later after storage in the silo) in a hammer mill. Powdered sugar is produced in different particle sizes. Powdered products have particular specifications regarding the particle size and amount of anticaking agent added to them. An anticaking agent, such as starch, is mixed with the powdered sugar during milling to prevent lumping (caking). (The finer the powdered sugar, the larger the amount of starch is added to prevent caking.) Usually, powdered sugar is tested for

- Particle size
- Wheat starch
- Maltodextrin

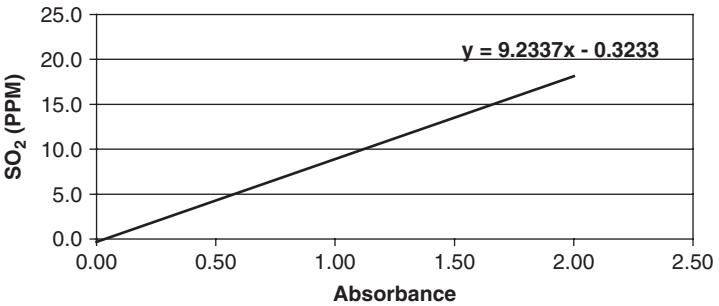


Figure 11.21 Relation between absorbance and SO_2 content

Particle Size of Powdered Sugar

Three types of powdered sugar are manufactured: 6X, 10X, and 12X (*the higher the number preceding the X, the finer the particles*), each having its specification. To test powdered sugar for particle size, use the following procedure:

1. Weigh a clean and empty 200-screen and record its mass (M_1).
2. Place the screen on the bench-top sieve shaker (Ro-Tap sieve shaker).
3. Put the weighing dish on the balance, zero the balance, and weigh 20.0 g sugar.
4. Empty the sugar from the weighing dish onto the screen.
5. Cover the screen with the top lid, set it on the shaker, and shake for 5 minutes.
6. Remove the screen from the shaker, reweigh it with the sugar, and record the mass (M_2).

Calculate the percentage of powdered sugar passing through the screen as follows:

$$\% \text{ On} = (M_2 - M_1) \times 5$$

$$\% \text{ Pass} = 100 - \% \text{ On}$$

Wheat Starch in Powdered Sugar

Wheat starch, unlike cornstarch, is not dissolved in cold water. Testing powdered sugar to determine wheat starch is based on the dissolving property of the sugar in water and the precipitation property of wheat starch in cold water. The following equipment is required for running the test:

- Analytical balance with readability to 0.0001 g
- Balance with readability to 0.01 g
- Microwave oven
- Oven with a temperature range of 30 to 200°C
- Vacuum pump with about 1.4 bar (20 lb) pressure
- Filtering flask and tubes for conducting a vacuum filtration
- Buchner funnel, 9.0 cm in diameter
- Stirrer
- Magnet for stirring
- Stirring bar retriever
- 250-ml flask
- Desicator
- Filter paper of 9.0 cm diameter (Whatman #2 or the equivalent)
- Timer
- Glass dish

For testing powdered sugar for **wheat starch content**, proceed as follows:

1. Put the filter paper into the oven at 110°C and dry it for 30 minutes.
2. Take the filter paper out of the oven and put it in the desicator for 10 minutes to cool it down.
3. Weigh the filter paper on the analytical balance and note its empty mass (M_1)
4. Put the filter paper into the Buchner funnel.
5. Weigh 20.00 g of powdered sugar.



Figure 11.22 Relation between polarimetric reading and cornstarch content

6. Put the magnet into a 250-ml flask and transfer the powdered sugar into the flask.
7. Add about 200 ml cold DI water to the flask and stir it for about 10 minutes to dissolve the sugar.
8. Use a retriever to take the magnet out of the flask and rinse it with a little of DI water.
9. Filter the solution through the funnel, which was prepared under step 4. After all solution is used, rinse the filter paper twice with DI water.
10. Take the filter out of the funnel, put it into the glass disk, and dry it in the microwave at a medium setting for 7 minutes (or in the oven for 2 hours at 105°C for 1 hour).
11. Take the filter from the microwave (or oven) and put it in the desicator for 10 minutes to cool.
12. Weigh it again on the analytical balance and record the mass (M_2).

Calculate the percentage of starch as follows:

$$\% \text{ Starch} = (M_2 - M_1) \times 5$$

Maltodextrin in Powdered Sugar

Maltodextrin is a **corn-based** starch that dissolves in cold water. To determine the amount of maltodextrin in powdered sugar, we use the **polarimetric method** as follows:

1. Weigh 26.0 g of powdered sugar and transfer it into a 200-ml Kohlrausch flask.
2. Add cool DI water and a magnet to the flask and stir to dissolve.
3. Take out the magnet and rinse it into the flask.
4. Fill to the mark with cool DI water.
5. Filter through medium porosity (almost 10-μm pore size) filter paper.
6. Rinse a polarimeter tube (200-mm polarimeter tube) twice with clear **filtrate** (the liquid collected during filtration) and fill the tube again.
7. Set the polarimeter to 0, place the tube in polarimeter, and record the reading when the polarimeter has stabilized.
8. Determine the % maltodextrin in the sample by polarimeter and by using a standard chart such as that shown in Figure 11.22. To make the standard graph, prepare a series of starch solutions containing 2 to 15% starch and polarize them on the polarimeter.

CHAPTER
11

SECTION *7*

SPECIAL METHODS OF ANALYSIS

SUBSECTIONS

- True Sucrose in Molasses by Inversion Method
- Invert Sugar in Different Products
- Microbiological Tests on Sugar
- Effective Alkalinity in First-Carb Juice
- Optimum Alkalinity in First-Carb Juice
- Dextran in First-Carb Juice
- High-Performance Liquid Chromatography

Special analyses are those tests that are not carried out routinely on a daily basis in the sugar lab. Some special tests, such as microbiological and invert sugar analyses on sugar, and invert sugar analyses on molasses, are usually carried out weekly on composite samples. Other special tests, such as optimum alkalinity, are carried out only occasionally on an as-needed basis.

TRUE SUCROSE IN MOLASSES BY INVERSION METHOD

The **inversion** (double-polarization) **method** is used to determine the true sucrose content. The polarimetric method can give correct results only when no optically active substances, except sucrose, are present in the sample. But besides sucrose, beet-juice samples contain at least three optically active sugars: raffinose (which has a positive effect on polarization), glucose (which has a positive effect), and fructose (which has a negative effect). For complete accuracy, the inversion method is used for some samples, such as determining true sucrose in molasses.

Generally, the inversion method consists of the following steps:

1. Converting sucrose to invert sugar by acid
2. Reading the polarimetric values of the sample before and after the inversion
3. Relating these values to calculate the true sucrose content

Several methods are used for inversion, including the Fehling, Muller, Clarget, and Berlin Institute methods. (Most such methods bear the author's or the institution's name.) The Berlin Institute method is explained here.

Beet-Sugar Handbook, by Mosen Asadi
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Sample Preparation

The molasses sample dry substance is adjusted as follows:

1. Double-dilute the sample by weighing 50.0 g of sample in a beaker and adding DI water to get a final mass of 100.0 g (see the Notes).
2. Stir the sample first with a glass rod and then with a magnetic stirrer until the sample is completely dissolved.
3. Cool the sample in a water bath to the standard temperature (20°C).
4. Apply the sample on the refractometer.
5. Record the reading for *RDS* (refractometric dry substance).

$$\%\ RDS = \text{Reading} \times 2$$

6. Keep the double-diluted sample for making the half-normal solution.

Half-Normal Solution

A half-normal solution of the sample is prepared as follows:

1. Make a half-normal solution by dissolving 52.0 g of double-diluted sample in a 200-ml flask.
2. Fill to the mark with distilled water at exactly 20°C.

Direct Polarization (DP)

The half-normal solution is polarized directly (without inversion) as follows:

1. Put 50.0 ml of the half normal solution into a 100-ml Kohlrausch flask.
2. Add clarifying reagents. For molasses, add 5 ml of **Reagent 1** (20% aluminum chloride solution) and 5 ml of **Reagent 2** (20% ammonium hydroxide solution), and mix it well.
3. Dilute to the mark with DI water.
4. Filter the sample into a beaker and return the first unclear portion (about the first 10 ml) of the filtrate back to the funnel. Use 18-cm Whatman #4 filter paper or the equivalent for filtration.
5. Rinse a polarimeter tube twice with the sample and fill the tube again.
6. Set the polarimeter to 0, place the tube in the polarimeter, and record the reading when the polarimeter has stabilized. This reading is called **direct polarization** (DP) because it is measured before inversion.

When a 100-mm polarimeter tube is used, the percentage of direct polarization (*DP*) is

$$\%\ DP = \text{Polarmetric reading} \times 8$$

Invert Polarization (IP)

The half-normal solution is polarized after inversion as follows:

1. Put 50.0 ml of the half-normal solution into a 100-ml flask.
2. Slowly add 10 ml of diluted hydrochloric solution (see Notes) and mix it well.
3. Place the flask in a hot-water bath (60°C) for exactly 10 minutes. Swirl the flask to bring the solution to temperature as soon as possible.
4. Cool the sample to 20°C, and dilute it to volume.

5. Rinse a polarimeter tube twice with the sample and fill the tube again.
6. Set the polarimeter to 0, place the tube in the polarimeter, and record the reading when the polarimeter has stabilized. This reading is called **invert polarization** (*IP*) because it is measured after inversion.

When a 100-mm polarimeter tube is used, the % *IP* is

$$\%\ IP = \text{Polarimetric reading} \times 8$$

Calculations

Calculate the results as follows:

$$\%\ \text{True sucrose} = \frac{0.512DP - IP}{0.839}$$

$$\%\ \text{True purity} = \frac{\%\ \text{True sucrose}}{\%\ RDS} \times 100$$

$$\%\ \text{Raffinose} = \frac{0.33DP + IP}{1.563}$$

$$\%\ \text{Raffinose}/RDS = \frac{\%\ \text{Raffinose}}{\%\ RDS} \times 100$$

EXAMPLE 10

Given:

Sample of molasses with the following results:

DP	50.4
IP	–14.2
RDS	81.5

Calculate:

True sucrose

True purity

% Raffinose

% Raffinose/*RDS*

$$\%\ \text{True sucrose} = \frac{0.512DP - IP}{0.839} = \frac{0.512 \times 50.4 - (-14.2)}{0.839} = 47.68$$

$$\%\ \text{True purity} = \frac{\%\ \text{True sucrose}}{\%\ RDS} \times 100 = \frac{47.68}{81.5} \times 100 = 58.5$$

$$\%\ \text{Raffinose} = \frac{0.33DP + IP}{1.563} = \frac{0.323 \times 50.4 - 14.4}{1.563} = 1.6$$

$$\%\ \text{Raffinose}/RDS = \frac{\%\ \text{Raffinose}}{\%\ RDS} \times 100 = \frac{1.55}{81.5} \times 100 = 1.9$$

NOTES

- **The molasses sample should be well mixed. If the molasses is too viscous for mixing at room temperature, put the sample in a closed container and put it in a water bath with a temperature of about 60°C for an hour before performing the analysis.**
- **To make a diluted hydrochloric acid solution, put 100 ml of hydrochloric acid (HCl) into a cylinder and dilute it to 24.8 *RDS*. This is approximately 6 parts of HCl to 5 parts water.**
- **Time and temperature are important in the inversion test. Both conditions must be correctly followed to obtain accurate results.**

INVERT SUGAR IN DIFFERENT PRODUCTS

Invert sugar refers to a mixture of equal amounts of glucose (dextrose) and fructose (levulose) resulting from the inversion of sucrose. Several methods for determining invert sugar are available, among them the **Berlin Institute method**, which is used in sugar products containing not more than 10% invert sugar in the presence of sucrose. The Berlin method is described here.

Principle

To the sample containing invert sugar (e.g. diffusion juice or molasses), Muller's solution is added to prepare a mixture with a pH of about 10.4 (relatively low in comparison with other methods). Invert sugar, which is a reducing sugar, reacts with cupric ions (in an alkaline solution) to precipitate cuprous oxide. Then, the liquid is acidified and an excess of iodine solution is added. When all the cuprous oxide has reacted with the iodine, the excess iodine is back-titrated with sodium thiosulfate, using starch as an indicator. Under the controlled conditions, the amount of copper reduced is proportional to the amount of invert sugar in the sample.

$$2C_6H_{12}O_6 + 4Cu(OH)_2 \rightarrow 2CH_2OH(CHOH)_4COOH + 2Cu_2O\downarrow + 4H_2O$$

Reagents

The following reagents are needed:

- Muller's solution
- Acetic acid solution, 5 *N*
- Iodine solution, 0.025 *N*
- Sodium thiosulfate solution, 0.025 *N*
- Starch indicator solution

Prepare **Muller's solution** as follows:

1. Dissolve 35 g of copper sulfate pentahydrate ($CuSO_4.5H_2O$) in 100 ml boiling DI water.
2. In another beaker, dissolve 173 g of sodium potassium tartrate tetrahydrate ($NaKC_4H_4O_6.4H_2O$) and 68 g of sodium carbonate (Na_2CO_3) in about 500 ml boiling DI water.
3. After cooling, pour both solutions into a 1-L flask and add DI water up to mark on the flask, and mix the solutions well.
4. Add a spoonful of activated charcoal and shake the flask vigorously.

5. Filter the solution through a filter paper under vacuum. If cuprous oxide is precipitated, the solution has to be filtered again.

Prepare **acetic acid solution**, 5 *N* as follows:

Dilute 300 ml of 96% acetic acid with DI water to volume in a 1-L flask.

Prepared **iodine solution 0.025 *N*** is available from chemical suppliers. Alternatively, prepare it as follows:

1. Dissolve 10 g of pure potassium iodide (KI) in a 1-L flask and add the minimum amount of DI water necessary to dissolve it.
2. Add 4.5 g iodine (I_2) to the solution and make up to volume with DI water.

Prepared **thiosulfate solution 0.025 N** is available from chemical suppliers. Alternatively, prepare it as follows. This solution has to be checked against iodine solution using phenol as an indicator.

1. In a 1-L flask, dissolve 8.3 g $Na_2S_2O_3.5H_2O$ (molecular mass = 248.2) with warm DI water.
2. Add 1 ml of 1 *N* sodium hydroxide (4 g in 100 ml) to it for stabilization and make up to 1000 ml with DI water.

The **starch indicator** (1% stabilized starch indicator) for iodometric titration is available from chemical suppliers. Alternatively, prepare it as follows:

1. Make a thin paste of 1 g of soluble starch (corn starch) in cold DI water.
2. Add the paste to 100 ml boiling DI water. Cool and add 5 mg of mercuric iodide (HgI_2) to stabilize. (**Note:** Mercuric iodide is **poisonous**.)

Sample Preparation

Prepare the sample so that it does not contain more than 30 mg of invert sugar.

1. **Blank sample:** Mix 20 ml DI water and 10 ml Muller solution in a 2.5 by 15 cm test tube. Test tubes should have **a short piece of glass rod** in them to facilitate mixing.
2. **Beet, diffusion juice, and thin juice:** Mix 1.00 g sample with 20 ml DI water and 10 ml Muller solution in a test tube (amount of sample = 1.0 g) and mix it well.
3. **Thick juice, standard liquor, and extract:** Weigh 10 g and make it to 100 ml in a flask with DI water (10% solution). Take 5 ml of this solution and mix it with 20 ml DI water and 10 ml Muller solution in a test tube (amount of sample = 0.5 g).
4. **Molasses:** Weigh 10 g and make it to 100 ml in a flask with DI water (10% solution). Take 1 ml of this solution and mix it with 20 ml DI water and 10 ml Muller solution in a test tube (amount of sample = 0.1 g).
5. **Raw sugar:** Mix 1.0 g of raw sugar with 20 ml DI water and 10 ml Muller solution in a test tube (amount of sample = 1.0 g).
6. **White sugar:** Mix 10 g of sugar with 20 ml DI water and 10 ml Muller solution in a test tube (amount of sample = 10 g).
7. **Powdered sugar:** Mix 1.0 g of powder sugar with 20 ml DI water and 10 ml Muller solution in a test tube (amount of sample = 1.0 g) and mix it well.

Procedure

To determine invert sugar in the samples, proceed as follows:

1. Place the tubes (blank and samples) in a boiling water bath so that the solution in the tubes is just below the level of the boiling water. Care must be taken to ensure that no boiling water gets into the tubes. The reaction time should be exactly 10 minutes.
2. Remove the tubes from the boiling water bath and immerse them in a cold-water bath. After a few minutes, the contents of the tubes will be sufficiently cool to be titrated.
3. Add 5 ml of 5 *N* acetic acid against the tube wall, close to the liquid level, to neutralize sodium carbonate in the solution. Do not shake the tubes during this step.
4. Immediately, add an excess amount of iodine (20 ml) until the solution becomes green.
5. Mix and pour the content of the tube into a 125-ml Erlenmeyer flask. Rinse the tube twice with small amount of DI water and add the rinse water to the flask. Mix the solution well until the precipitate is completely dissolved.
6. Add 5 drops of starch indicator, and immediately titrate the excess iodine with sodium thiosulfate to the endpoint (light **blue** color). Record the ml of thiosulfate used for the blank and for each sample.

Calculation

Each ml of excess of iodine solution reacted with thiosulfate is equivalent to 1 mg invert sugar. So the amount of invert sugar on dry substance is calculated according to the following formula:

$$\%\ \text{Invert}/RDS = \frac{\text{ml Thiosulfate}_{\text{Blank}} - \text{ml Thiosulfate}_{\text{Sample}} - 0.2}{\text{g Sample} \times RDS_{\text{Sample}} \times 1000} \times 100 \times 100$$

$$\%\ \text{Invert}/RDS = \frac{\text{ml Thiosulfate}_{\text{Blank}} - \text{ml Thiosulfate}_{\text{Sample}} - 0.2}{\text{g Sample} \times RDS} \times 10$$

The number 0.2 in the numerator is the sucrose correction due to its reducing action (0.2 ml iodine solution per gram of sucrose).

EXAMPLE 11

Given:
When testing a molasses sample for invert-sugar content, the following readings were found:

ml of thiosulfate for blank	20.0
ml of thiosulfate for sample	18.4
Sample *RDS*	81.6
Amount of sample	0.1

Calculate:
% Invert sugar/*RDS*

$$\%\ \text{Invert}/RDS = \frac{20.0 - 18.4 - 0.2}{0.1 \times 81.6} \times 10 = 1.71$$

EXAMPLE 12

Given:
When testing powdered sugar with maltodextrin, the following readings were found:

ml of thiosulfate for blank	20.0
ml of thiosulfate for sample	3.8
Sample *RDS*	100
Amount of sample	1.0

Calculate:
% Invert sugar/*RDS*

$$\%\ \text{Invert}/RDS = \frac{20.0 - 3.8 - 0.2}{1.0 \times 100} \times 10 = 1.6$$

Because maltodextrin contains only 15.0% of invert sugar, the result of the test should be multiplied by 100/15 = 6.66 to find the whole amount of starch in the sample.

$$1.6 \times 6.66 = 10.7\%\ \text{Starch}$$

MICROBIOLOGICAL TESTS ON SUGAR

Sugar produced under insufficient hygiene conditions may carry some microorganisms that can spoil canned foods and carbonated beverages. Microbiological tests are usually performed weekly on composite samples of sugar. Sugar is usually tested for yeasts and molds, and mesophilic and thermophilic bacteria, which belong to an aerobic group (organisms that grow in the presence of oxygen).

The method described here complies with the ICUMSA method. An aliquot of sugar or liquid sugar in sterile water is mixed with special nutrient media and incubated. After incubation, the developed colonies are counted. The results are reported as colonies per 10 g of sugar. (Note: The test should be performed hygienically and by an experienced analyst.)

Equipment

The following equipment is required:

- One 100-ml Erlenmeyer flask, sterilized
- Two 250-ml Erlenmeyer flasks, sterilized
- One 5-ml pipet, sterilized
- Two magnets for mixing agars, sterilized
- Four Petri dishes, sterilized if not disposable
- Three porcelain dishes for weighing the sugar sample
- An oven maintained at 30°C

- An oven maintained at 120°C for sterilization of culture media
- An autoclave (sterilizer) for sterilizing dishes and liquids

Sterilization

Consider the following recommendations:

- The lab bench must be cleaned thoroughly before microbiological work is started.
- All apparatus should be cleaned and sterilized in an **autoclave** (sterilizer) for 15 minutes or in a hot air oven at 160°C for two hours.
- DI water can be sterilized by preparing it in a 1-L screw-capped Pyrex bottle followed by autoclaving for 15 minutes.
- The sugar sample should be taken from a suitable and noncontaminated place and should be kept in sterile container.
- The sugar should be weighed directly into a sterile flask using a flamed spatula.
- The necks of pipets and flasks should be flamed in a Bunsen burner to kill out organisms.
- Pipets should not be allowed to touch the bench until the operation is complete.

Preparation of Media

Three media are required:

- Medium for yeast and mold
- Medium for mesophilic bacteria
- Medium for thermophilic bacteria

The **medium to detect yeast and mold** is available as **dextrose agar**. To prepare this medium, proceed as follows:

1. Add 9.5 g of dextrose agar medium in 250 ml of sterile DI water.
2. Mix thoroughly by heating and frequent agitation, and boil for about 1 minute to completely dissolve the powder.
3. Sterilize the medium by autoclaving at 120°C for 15 minutes.

Preferably, the medium should be cooled and used at once. If the medium is allowed to solidify after autoclaving, it may be remelted once again by heating. Avoid overheating at all times.

(Note that ready media are available, eliminating the need for preparation: e.g., Redigel potato dextrose, available in the United States.) When using commercial media, follow the instructions that come with the supply.

The **medium to detect mesophilic bacteria** is available as **nutrient agar**. To prepare this medium, proceed as follows:

1. Add 5.75 g of nutrient agar in 250 ml of sterile DI water, and mix thoroughly.
2. Heat the mixture with frequent agitation, and boil for 1 minute to completely dissolve the agar.
3. Sterilize the solution by autoclaving at 120°C for 15 minutes.

The medium to detect thermophilic bacteria is available as **Shapton agar**. To prepare this medium, proceed as follows:

1. Add 5.75 g of *Shapton* agar in 250 ml of sterile DI water, and mix thoroughly.
2. Heat the mixture with frequent agitation, and boil for 1 minute to completely dissolve the agar.
3. Sterilize the solution by autoclaving at 120°C for 15 minutes.

Preparation of Sample

Dissolve 50.0 g sugar in sterile DI water and make up to 100 ml. In the case of liquid sugar, adjust the concentration to 50% m/m.

Determination

To determine **yeast** and **mold**, proceed as follows:

1. Mark two Petri dishes with **Y + M**, for yeast and mold.
2. Add 2.5 ml of the sample (contains 1.25 g sugar) into each dish.
3. Cover the samples with about 15 ml of medium.
4. Incubate them for 72 hours at 30°C.
5. Count the yeast and molds after the prescribed time.

Yeast colonies tend to grow as *small, shiny, white colonies with defined borders*. Mold colonies may be *green, white, gray, black, or other colors and most likely appear fuzzy*. The counts from both plates represent the bacteria in 2.5 g. The counts are multiplied by 4 to express the result as colonies per 10 g sugar.

(Note: Some additional bacterial growth with different shapes may occur. These should be ignored in recording the counts.)

To determine **mesophilic** bacteria, proceed as follows:

1. Mark two Petri dishes with **M**, for mesophilic.
2. Add 2.5 ml of the sample (this contains 1.25 g sugar) into each dish.
3. Cover samples with about 15 ml of agar.
4. Incubate them for 48 hours at 30°C.
5. Count colonies after the prescribed time.

Combine the counts from two plates and multiply by 4 to find the colonies per 10 g sugar.

To determine **thermophilic** bacteria, proceed as follows:

1. Steam 50 ml of the sample in a sterile graduated flask at atmospheric pressure for 20 minutes to kill everything but the spores.
2. Cool the sample to room temperature rapidly in a cold water bath.
3. Add sterile DI water to the 50 ml line of the flask.
4. Mark two Petri dishes with **T**, for thermophilic.
5. Add 2.5 ml (this contains 1.25 g sugar) of the sample into each dish.
6. Cover the samples with about 15 ml of agar.

7. Incubate them for 48 hours at 47°C in an inverted position.
8. Count the colonies after the prescribed time.

Combine the counts from two plates and multiply by 4 to find the colonies per 10 g sugar.

NOTE

10 g sugar sample should not contain more than 10 yeasts, 10 molds, and 200 mesophilic or thermophilic colonies.

EFFECTIVE ALKALINITY IN FIRST-CARB JUICE

Effective alkalinity is the amount of dissolved K and Na carbonates and bicarbonates in the juice. To determine the effective alkalinity, two tests should be performed:

- Alkalinity test
- Hardness test

Alkalinity Test

Obtain a sample of **first-carb juice** and **filter it right away**. Otherwise, precipitated hardness substances (limesalts) start to dissolve as the temperature decreases. Proceed then as follows:

1. Cool the filtered sample to the standard temperature, and transfer 10 ml of the sample into a 100-ml flask.
2. Add about 20 ml DI water to lighten the color of the sample.
3. Put a pH electrode into the flask and titrate with Reagent 6 (0.036 *N* sulfuric acid solution) to an endpoint of 9.2 pH.

$$\text{Alkalinity} = \text{ml}_{\text{Acid}} \times 0.01\,\text{g}/100\,\text{ml}$$

Hardness Test

To test the sample for **hardness content**, use the following procedure:

1. Transfer 10 ml of the filtered sample into a casserole dish, add about 20 ml DI water, 2 to 3 shakes of **Reagent 8** (hardness indicator) and 2 ml of **Reagent 9** (hardness buffer solution) for pH adjustment, and mix well.
2. Titrate with **Reagent 10** (hardness titrant) to a **blue** color. 1 ml of hardness titrant (0.036 EDTA) equals 0.001 g CaO:

$$\text{Hardness} = \text{ml}_{\text{Titrant}} \times 0.001 \times \frac{10}{100} = \text{ml}_{\text{Titrant}} \times 0.01\,\text{g}/100\,\text{ml}$$

The effective alkalinity is calculated as

$$\text{Effective alkalinity} = \text{Alkalinity (titration to pH} = 9.2) - \text{Hardness}$$

NOTE

The effective alkalinity of the first-carb juice should be between 0.005 and 0.010% CaO on juice. If the alkalinity is lower, you may try to restore the alkalinity by adding either soda ash (Na_2CO_3) or magnesium oxide (MgO, known as Magox) to the final carbonation juice before the second-carb filtration.

OPTIMUM ALKALINITY IN FIRST-CARB JUICE

The **optimum-alkalinity** test is performed to find out whether or not the juice-purification operation has achieved maximum removal of hardness (the dissolved salts of Ca and Mg) during the first-carbonation step. At optimum alkalinity, the juice has the lowest amount of hardness (limesalts). This test is performed on **filtered first-carb** juice to determine the optimum alkalinity of the juice entering the second carbonation. To determine optimum alkalinity, proceed as follows:

1. Prepare 4 beakers (350 ml) and put about 200 ml of filtered first-carb juice in each.
2. Insert a pH electrode into the first beaker and start adding CO_2 gas to pH = 9.0.
3. Gas the other three beakers to 8.9, 8.8, and 8.6, respectively.
4. Filter the samples immediately (be careful because precipitated hardness starts to dissolve as the juice cools), and run total alkalinity and hardness tests on all samples.
5. Plot the results on graph paper and find the optimum alkalinity, which is equal to the lowest hardness content.

DEXTRAN IN FIRST-CARB JUICE

Dextran in first-carb juice sample is tested by a **haze method**. By adding alcohol to the sample containing **dextran** (a polysaccharide), you produce **dextran haze**, which can be read on a spectrophotometer. To determine the dextran content, proceed as follows:

1. Take about 100 ml sample of the first-carb juice and filter it.
2. Acidify the sample with concentrated HCl to pH = 2.
3. Filter the sample.
4. Mix 25 ml of the filtered sample with 25 ml of DI water.
5. Mix another 25 ml of the filtered sample with 25 ml of 99.9% ethanol.
6. Zero the spectrophotometer with DI water.
7. Measure the absorbance (A) of the sample in step 4 in a 10-mm tube at 760 ($A1$).
8. Measure the absorbance of the sample in step 5 ($A2$).
9. Add the difference between the absorbances of $A1$ and $A2$ (+ or –) to 0.035, and then divide the value by 0.0008383. The result is mg dextran per liter of juice (ppm).

NOTES

- **At higher concentrations of dextran, you can dilute the sample two or four times with DI water before the acid treatment and then multiply the result by 2 or 4.**
- **To obtain coefficients from a standard curve for the dextran, plot the absorbance of standard dextran solutions. The values used in step 9 (0.035 and 0.0008383) were derived by this method.**
- **In the haze method, other polysaccharides that also form an alcohol haze increase the test result, compared to an accurate dextran test (by the Roberts method or by HPLC).**

HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY

High-performance liquid chromatography (HPLC) is a technique for separating a sample into its components and then measuring the amount of the components by a detector. The

HPLC technique is one of the best ways to analyze the amount of betaine fraction in the process of **molasses desugaring by chromatographic process** (MDC process). In general, the HPLC can be used whenever the sample can be dissolved in a liquid.

The HPLC system consists of a **stationary** phase and a **mobile** phase. The stationary phase (ion-exchange resin) is in a column, and the sample (mobile phase) is fed by a pump through the resin. Then, a **solvent** (eluent) is used to wash the components from the resin. As the sample moves through the resin, its molecules separate and move downward at different rates. The rate of movement of the individual molecules depends on the chemical properties and size of the molecules, which is called the **affinity of the molecules** toward the resin. In other words, one type of molecule might have a tendency to attach to the surface of the resin and another molecule might dissolve in the solution and move much faster through the resin. Separation is obtained when one molecule is **behind in movement** enough to prevent overlap with the peak of an adjacent molecule.

The HPLC technique depends on the equipment, the type of chromatographic column, and other devices of the system, which are different from one laboratory to another. The principle of HPLC described here uses general terms with no specific brand of instrument.

Following are the **main components of a chromatograph**:

- Pump
- Column
- Integrator
- Guard column
- Sampling valve
- Connecting tubing
- Detector: For detecting the state of solutes in the mobile phase (the refractive index detector is the most common for sugar analyses)

Following are the reagents needed for performing an **HPLC test** on a **betaine** sample:

- Betaine anhydrous ($C_5H_{11}NO_2$): usually a 0.5 mg/ml betaine solution for standardization
- Sodium sulfate (Na_2SO_4): usually a 0.015 *M* solution used as the eluent
- Acetonitrile (CH_3CN): 30% solution for cleaning column
- Sodium phosphate dibasic (Na_2HPO_4): 0.02 *M* solution for column regeneration
- Double-ionized water: for preparing reagents and diluting the sample

The betaine sample should be diluted to a 5% solution. The following parameters should be maintained:

- Flow rate to chromatograph: 0.6 ml/min
- Column temperature: 85°C
- Detector temperature: 35°C

For **calculation**, the amount of betaine is set up as a standard linear regression curve versus peak area. The amount of betaine (in mg/ml) is calculated using a standard graph. The result is then calculated on gram betaine in 100 g *DS* (dry substance).

CHAPTER
11

SECTION 8

MOLASSES-DESUGARING METHODS OF ANALYSIS

SUBSECTIONS

- Molasses Tests
- Diluted Molasses Tests
- Betaine Tests
- Raffinate Tests
- Extract Tests

This section describes the analytical tests related to **molasses desugaring by chromatographic process** (MDC process). The MDC process operates in two- or three-fraction mode. In two-fraction mode, it produces **extract** (the product of the MDC process with high purity) and **raffinate** (the byproduct of the process with low purity and high ash content). Note: Raffinate is also known as CMS (concentrated molasses solids). If the MDC process is set up to operate in three-fraction mode, the fractions are extract, raffinate, and betaine. The following procedures explain the analysis in three-fraction operation.

The following tests are provided by the desugaring analyst:

- Molasses (two times per shift)
- Diluted molasses (three times per shift)
- Betaine (one time per shift)
- Concentrated betaine (three times per shift)
- Raffinate (one time per shift)
- Concentrated raffinate (three times per shift)
- Extract (one time per shift)
- Concentrated extract (three times per shift)

NOTE

In the ARI (Amalgamated Research Inc.) desugaring method, the extract fraction is collected after passing two sets of chromatographic loops. The product of the first loop is called an upgrade, which is first concentrated in the evaporators and then enters the second loop. The procedures for testing the upgrade fraction and the interval of the tests are similar to that for testing the raffinate.

Beet-Sugar Handbook, by Mosen Asadi
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Day: Date: Molasses: Sheet 3 (Desugarization)

	Feed							Upgrade									Betaine					Raffinate					Extract							
	Heavy			Diluted				Before				Concentrate					Before		Concentrate			Before		Conc. (CMS)			Before				Concentrate			
	RDS	Purity	Color	RDS	pH	Salts	Sed.	RDS	Purity	Colorp	H	RDS	Purity	Colorp	H	Sed	RDS	Purity	RDS	Purity	pH	RDS	Purity	RDS	Purity	pH	RDS	Purity	Color	pH	RDS	Purity	Color	pH
7am																																		
8																																		
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Avg.																																		

Figure 11.23 Desugaring lab sheet

MOLASSES TESTS

Molasses, used as the feed for the MDC process, is analyzed for the following tests:

- **RDS**
- Sugar content
- Color

To test a **molasses** sample for ***RDS***, use the following procedure:

1. Double-dilute the sample by weighing 50.0 g of the sample in a beaker and adding hot DI water to give a final mass of 100.0 g.
2. Stir the sample first with a glass rod and then with a magnetic stirrer until the sample is completely mixed (use a retriever to take the magnet out of the flask).
3. Cool the sample in the water bath to the standard temperature (20°C).
4. Apply the sample on the refractometer and record the reading.

$$\%\ RDS = \text{Reading} \times 2$$

5. Keep the rest of the sample for next test (the sugar test).

To test a **molasses** sample for **sugar content**, use the following procedure:

1. Weigh 26.00 g of the cold sample left from the double-diluted solution (step 5 of the *RDS* test) into a weighing dish.
2. Transfer into a 200-ml Kohlrausch flask using a plastic wash bottle with DI water.
3. Add 5 ml of **Reagent 1** (20% aluminum chloride solution) and 5 ml of **Reagent 2** (20% ammonium hydroxide solution) and mix it well.
4. If the sample is not cold, cool it to the standard temperature (20°C).
5. Use a wash bottle to add DI water to the neck of the flask.
6. Fill with DI water exactly to the mark of the flask using a dropper. Pay attention that the bottom of meniscus and the line correspond exactly.
7. Stopper the flask and mix it well by inverting and shaking, allowing bubbles to escape from the flask.
8. Let it set for about 1 minute.
9. Filter into a beaker and return the first unclear portion (about the first 10 ml) of the filtrate back to the funnel. Use 18-cm Whatman #4 filter paper or the equivalent for filtration.
10. Rinse a polarimeter tube twice with clear filtrate and fill it again.
11. Set the polarimeter to 0, place the tube in the polarimeter, and record the reading when the polarimeter has stabilized.

When a 100-mm polarimeter tube is used, the percentage of sugar is

$$\%\ \text{Sugar} = \text{Polarimetric reading} \times 8$$

Calculate the **purity** (*P*) according to the purity equation:

$$\%\ P = \frac{\%\ \text{Sugar}}{\%\ RDS} \times 100$$

To test the **molasses** for **color**, follow the procedure listed under Standard Liquor Analysis subsection in the Sugar-End Methods of Analysis section.

DILUTED MOLASSES TESTS

Before molasses is fed to the separator, it is diluted to 60 to 70% *DS* (depending on the type of the desugaring process). The diluted molasses should be analyzed for the following tests:

- **RDS**
- PH
- Hardness (limesalts)
- Sediment (suspended solids)

To test a **diluted-molasses** sample for ***RDS***, use the following procedure:

1. Cool the sample to the standard temperature (20°C).
2. Apply the sample on the refractometer
3. Read it directly for the % *RDS*.

To test a **molasses** sample for **pH**, use the following procedure:

1. Rinse the electrode of the pH-meter and beaker with a portion of the cold sample (20°C).
2. Take sufficient sample in the beaker and immerse the electrode in the sample.
3. Read the **pH** when the reading has stabilized.

To test a **diluted-molasses** sample for **hardness content**, use the following procedure:

1. Prepare 100 ml solution of about 2% *RDS* by using the following procedure:

$$\text{Mass}_{\text{Syrup}} = \frac{RDS_{\text{Required}}}{RDS_{\text{Syrup}}} \times \text{Mass}_{\text{Required}}$$

$$\text{Mass}_{\text{Water}} = \text{Mass}_{\text{Required}} - \text{Mass}_{\text{Syrup}}$$

For example, if the *RDS* of molasses is 60.5, make 100 g of 2% *RDS* solution as follows:

$$\text{Mass}_{\text{Syrup}} = \frac{2.0}{60.5} \times 100 = 3.3\,\text{g Juice}$$

$$\text{Mass}_{\text{Water}} = 100 - 3.3 = 96.7\,\text{g Water}$$

Neither the volume nor the *RDS* has to be exact, since the *RDS* of the solution will be measured later (in step 3).

2. Mix the sample well and apply it on the refractometer.
3. Read the % **RDS** of the solution and record the reading.
4. Pipet 10 ml of the sample to a porcelain dish. If the color of the sample is still dark, add about 10 ml more DI water to reduce the intensity of the color.
5. Add 2 ml of **Reagent 8** (hardness buffer solution) and 2 to 3 shakes of **Reagent 9** (hardness indicator) to the dish to give a good **pink** color, and stir well to dissolve the powder.

6. Titrate with **Reagent 10** (hardness titrant) to the first color change and stop it. Proceed slowly (drop by drop) to the **blue** endpoint. (Note: The endpoint should not fade. 1 ml of titrant [0.036 EDTA] is equivalent to 1000 mg CaO.)

Calculate the hardness as follows:

$$\text{Hardness} = \frac{\text{ml}_{\text{Titrant}}}{\%\ RDS} \times 1000\,\text{mg CaO}/RDS$$

$$\text{Equivalent mass} = \frac{\text{Molecular mass (CaO)}}{\text{Number of charges (CaO)}} = \frac{56}{2} = 28$$

or

$$\text{Hardness} = \frac{\text{ml}_{\text{Titrant}}}{\%\ RDS} \times \frac{1000}{28} = \frac{\text{ml}_{\text{Titrant}}}{\%\ RDS} \times 35.7\,mEq\,\text{CaO}/RDS$$

NOTE

Because the desugaring process uses soft molasses (the molasses that contains less than 3 *mEq* CaO/100 *DS*), you must titrate the sample with a dropper instead of a buret (called drop titration). In this case, the formula is divided by 20 because each ml equals almost 20 drops.

$$\textbf{Hardness} = \frac{\textbf{Drops}_{\text{Titrant}}}{\textbf{\textit{RDS}}} \times \frac{35.7}{20} = \frac{\textbf{Drop}_{\text{Titrant}}}{\textbf{\textit{RDS}}} \times \textbf{1.8}\ \textbf{\textit{mEq}}\ \textbf{CaO}/\textbf{\textit{RDS}}$$

Sediment (suspended solids) content in the feed to separators is very important to the separation process because feed containing high amounts of suspended solids decreases the efficiency of the separation and finally can foul the resin. To test a **diluted-molasses sample** for **sediment**, proceed as follows:

1. Weigh a 3-cm filter disk with medium porosity (almost 10 µm pore size) on an analytical balance with a readability of 0.0001 g, and note the weight (M_1).
2. Transfer the disk, with the help of a tweezers, to a Buchner funnel (made for the 3-cm disk) connected to a vacuum.
3. Wet the disk with DI water so that the disk fits tightly around the sides of the funnel, using the vacuum to pull the water through the disk.
4. Pour a measured amount of hot sample (100 to 250 ml depending on the sample's turbidity), using a graduated cylinder.
5. Rinse the graduated cylinder with hot DI water and pour the wash water into the funnel.
6. Rinse the funnel with about 50 ml of hot DI water.
7. Allow the disk to drain for 2 minutes under the vacuum.
8. Stop the vacuum, remove the disk with a tweezers, and place it in a Petri dish.
9. Dry in an oven at 105°C for 3 hours.
10. Remove the disk from the oven and cool it in a desicator for 10 minutes.
11. Weigh the disk on the analytical balance (M_2).

Calculate the suspended solids (*SS*) as ppm:

$$SS = \frac{M_2}{M_1} \times \frac{10^6}{\text{ml}_{\text{Sample}}}$$

NOTE

In ordinary operational work, the weighing and drying of the disk after filtration may be omitted from the procedure to save time. In this case, the disk is compared with standard disks containing 1, 2, and 3 ppm of suspended solids and marked accordingly.

BETAINE TESTS

Betaine is one of the by-products of the molasses-desugaring process. This fraction has low concentration (around 10% *RDS*) when it comes off the separator, so it is concentrated in the evaporator to about 60% *RDS* for sale. You should test the betaine sample, taken before evaporation, for dry-substance (*DS*) content. The sample, taken after evaporation, is tested for *DS* content, sugar content, pH, and betaine content (not routinely). Because of their similarity, only the procedures for concentrated betaine are explained here. The betaine sample is analyzed for the following tests:

- **RDS**
- Sugar content
- PH
- Betaine content

To test a **betaine sample** for ***RDS***, use the following procedure:

1. Cool the sample to the standard temperature (20°C).
2. Apply the sample on the refractometer.
3. Read it directly for the % *RDS*.

To test a **betaine sample** for **sugar content**, use the following procedure:

1. Weigh 26.00 g of cold sample in a weighing dish.
2. Transfer into a 200-ml Kohlrausch flask by washing with DI water using a wash bottle.
3. Add 5 ml of **Reagent 1** (20% aluminum chloride solution) and 5 ml of **Reagent 2** (20% ammonium hydroxide solution).
4. Mix well by swirling.
5. Use a wash bottle to add DI water to the neck of the flask.
6. Raise exactly to the mark on the flask by applying the DI water with a dropper.
7. Stopper the flask and mix it well by shaking, allowing bubbles to escape from the flask.
8. Let it set for about 1 minute.
9. Filter into a beaker and return the first unclear portion (about the first 10 ml) of the filtrate back to the funnel. Use 18-cm Whatman #4 filter paper or equivalent for filtration.
10. Rinse a polarimeter tube twice with the sample and fill the tube again.
11. Set the polarimeter to 0, place the tube in the polarimeter, and record the reading when the polarimeter has stabilized.

When using a 100-mm polarimeter tube, the result is

$$\% \text{ Sugar} = \text{Polarimetric reading} \times 4$$

NOTE

If you have an unstable polarimetric reading, reduce the sample weight to half. Then, multiply the reading by 8 to find the % sugar.

Calculate the **purity** (*P*) according to the purity equation:

$$\% P = \frac{\% \text{ Sugar}}{\% RDS} \times 100$$

The procedure for the **pH** test for a **betaine sample** is the same as that explained in the Diluted Molasses Tests section.

The **betaine content** can be tested weekly on a composite sample because it has to be analyzed by the time-consuming **HPLC** method. HPLC is one of the best ways for determining betaine in betaine samples. For more information, see the Special Methods of Analysis section.

NOTE

Betaine can be analyzed by an outside laboratory contractor equipped with HPLC.

RAFFINATE TESTS

The **raffinate** (some call it CMS for concentrated molasses solids) fraction contains most of the ashes existing in the molasses and has a low concentration (around 10% *RDS*) when it comes off the loop, so it is concentrated in the evaporator to about 60% *RDS* to be stored for sale. You should test the raffinate sample, taken before evaporation, for *RDS* content. The sample, taken after evaporation, is analyzed for the following tests

- **RDS**
- Sugar content
- pH

NOTE

For analyses of raffinate, proceed as previously explained in the Betaine Tests subsection.

EXTRACT TESTS

The **extract** fraction, obtained from the desugaring operation, contains most of the sugar present in the molasses, so its purity is high compared with the betaine and raffinate fractions. You should test the extract sample, taken before evaporation, for dry-substance (*DS*) content. Because of their similarity, only the procedures for a concentrated extract are explained here. The postevaporation sample is analyzed for the following tests:

- **RDS**
- Sugar content
- PH
- Color

To test an **extract sample** for ***RDS*** (refractometric dry substance), use the following procedure:

1. Cool the sample to the standard temperature (20°C).
2. Apply the sample on the refractometer.
3. Read it directly for the % *RDS*.

To test an **extract sample** for **sugar content**, use the following procedure:

1. Weigh 26.00 g of cold sample in a weighing dish.
2. Transfer into a 200-ml Kohlrausch flask by washing with DI water using a wash bottle.
3. Mix well by swirling.
4. If it is not cold, cool to the standard temperature (20°C)
5. Add DI water from a wash bottle to below the neck of the flask.
6. If foam is on the surface, add 2 drops of ether to clear the foam.
7. Raise the volume to the mark on the flask, applying DI water with a dropper.
8. Stopper the flask and mix it well by shaking, allowing bubbles to escape from the flask.
9. Rinse a polarimeter tube twice with the sample and fill the tube again.
10. Set the polarimeter to 0, place the tube in the polarimeter, and record reading when the polarimeter has stabilized.

When a 100-mm polarimeter tube is used, the result is

$$\%\ \text{Sugar} = \text{Polarimetric reading} \times 4$$

Calculate the **purity** (*P*) according to the purity equation:

$$\%\ P = \frac{\%\ \text{Sugar}}{\%\ RDS} \times 100$$

The procedure is the same as that explained in the Diluted Molasses Tests section.

To test an **extract sample** for **color**, use the following procedure:

1. Prepare 100 ml solution of about 5% *RDS* by using

$$\text{Mass}_{\text{Syrup}} = \frac{RDS_{\text{Required}}}{RDS_{\text{Syrup}}} \times \text{Mass}_{\text{Required}}$$

$$\text{Mass}_{\text{Water}} = \text{Mass}_{\text{Required}} - \text{Mass}_{\text{Syrup}}$$

For example, if the *RDS* of the extract is 58.5%, you can make 100 g of 10% *RDS* solution using

$$\text{Mass}_{\text{Juice}} = \frac{5.0}{70.2} \times 100 = 7.1\,\text{g Extract}$$

$$\text{Mass}_{\text{Water}} = 100 - 7.1 = 92.9\,\text{g Water}$$

2. Measure the % *RDS* of the prepared sample on the refractometer.
3. Fill a color tube (1.0 or 2.5 cm in diameter) with the sample.
4. Fill another tube with DI water as the **blank** (reference).
5. Adjust the spectrophotometer reading to 0 at 420 nm with the blank using transmittance (*T*) or absorbance (*A*) mode of the instrument.
6. Read the *T* or *A* of the sample.

Calculate the color expressed as ICUMSA 420 (**IU color**) as follows:

$$C = 10^6 \frac{A}{b \times RDS \times d}$$

When using the *T* mode:

$$C = 10^6 \frac{2 - \log T}{b \times RDS \times d}$$

b Cell path length (sample tube diameter), in mm
d Density, from Table A.1 in the Appendix or from *RDS*-density regression equation ($d = 0.0055 \times RDS + 0.9714$)

EXAMPLE 13

Given:

b	25.4
RDS	5.2
T	6.2

Calculate:
Color of the sample

$$d = 0.0055 \times RDS + 0.9714 = 0.0055 \times 5.2 + 0.9714 = 1.0$$

$$C = 10^6 \frac{2 - \log T}{b \times RDS \times d} = 10^6 \frac{2 - \log 6.2}{25.4 \times 5.2 \times 1.0} = 8790$$

CHAPTER
11

SECTION 9

ENVIRONMENTAL METHODS OF ANALYSIS

SUBSECTIONS

- Chemical Oxygen Demand
- Biochemical Oxygen Demand
- Solids in Wastewater
- Nitrogen in Wastewater

Over the last three decades, most governments in the world have established environmental protection organizations to protect the health of their people by protecting the environment. For example, the **Environmental Protection Agency** (EPA) was established in 1970 by the U.S. government for such purposes.

To comply with environmental regulations, sugar factories must have several permits, such as wastewater discharge, air pollution, odor, noise, and hazardous waste. Each permit has its own rules (discussed in Chapter 10), sampling, control analytical methods, and more. This section explains methods for analyzing wastewater in sugar factories.

CHEMICAL OXYGEN DEMAND

Chemical oxygen demand (COD) is a quick test for rough estimation of the amount of organic substances in wastewater. The **COD** test measures roughly the use of oxygen equal to the amount of *organic substances* present in the wastewater. (*The higher the presence of organic substances, the higher are the oxygen demands*.)

Principle

The oxygen in the wastewater is subjected to oxidation by a strong chemical oxidant, such as dichromate potassium. In this method, a measured extra quantity of potassium dichromate and acid sulfuric is added to the sample and the mixture is boiled. Part of the dichromate is consumed in the oxidation of the organic substances, and the COD can be determined colorimetrically by measuring the increase in the *green* color of the chromium (Cr) or the decrease in the yellow color of the dichromate (Cr_2O_7), or by titration of the remaining dichromate by the titrimetric method. The results are expressed in mg/L (ppm).

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Equipment

The following equipment is required for the COD test:

- COD reactor
- Digestion reagent vials (produced by scientific chemical suppliers). See the Notes for the range of vials.
- Vial adapter for the spectrophotometer
- Spectrophotometer (programmed for COD tests; e.g., the Hach model)

Procedure

To run a sample for COD, proceed as follows:

1. Turn on the COD reactor to preheat to 150°C.
2. Remove the cap from a COD digestion reagent vial. While holding the vial away from your body, carefully pipet 2 ml of sample into the vial (see the Notes).
3. Replace the cap and tighten carefully.
4. Holding by the cap, invert it several times to mix. CAUTION: The vial becomes very hot during mixing, so do not mix near your face.
5. Take another digestion reagent vial and pipet 2 ml DI water. Repeat steps 3 and 4. Use this vial as the **blank** (reference).
6. Place both vials in the preheated COD reactor. Let them set for 2 hours at 150°C.
7. Turn the reactor off after the heating time and allow the vials to cool to the point where they can be handled. Then place the vials in a cooling rack and allow them to cool to the touch.
8. Measure the COD of the sample on the spectrophotometer against the blank at 620 nm wavelengths (follow the instruction of the spectrophotometer for its operation).

NOTES

- **COD digestion reagent vials come in different ranges, for example, 150, 1500, and up to 15000 mg/l (ppm).**
- **If the COD is more than the vial range, a green color occurs in the reacted sample because all the dichromate has been used. If this is the case, take 10 ml of the sample in a 100-ml flask and bring to the mark with DI water. Mix well and pipet 2 ml of this solution into the new digestion reagent vial. Then multiply the reading from spectrophotometer by 10.**

BIOCHEMICAL OXYGEN DEMAND

Biochemical oxygen demand (BOD) is a test for measuring the amount of organic substances in wastewater by determining the amount of oxygen, required to biologically oxidize the organic substances in a sample over time (usually 5 days). Organic substances may be dissolved (like sugar) or suspended (as solids in sewage).

You calculate BOD by comparing the amount of **dissolved oxygen** (DO) left at the end of 5 days with the amount known to be present at the beginning (difference = amount consumed). The determination is made by diluting a measured quantity of wastewater with a mea-

sured quantity of dilution water. The **dilution water** is DI water saturated with oxygen to which is added an inorganic **nutrient buffer** (nutrient for the growth of bacteria).

Principal

The blank (100% dilution water) and diluted samples are first tested for **DO** and then incubated in the incubator at 20°C, in the dark, for 5 days. At the end of incubation period, another DO determination is made on the blank and each of the samples. The amount of oxygen consumed during the 5 days in relation to the volume of the sample is used to calculate the BOD.

Equipment

- BOD bottles (preferably numbered), 300-ml capacity with cap
- BOD incubator, controlled at 20°C
- pH meter with mV readability, such as Orion 720A or equivalent
- BOD electrode, such as Orion model 97-08 or equivalent
- Magnetic stirrer

Sampling

Samples are taken in glass-stopper bottles and are filled to overflowing so that, when the stopper is inserted, no air bubbles are trapped. (Air bubbles can furnish oxygen, which can react with the sample before it is diluted and placed in the water bath.)

BOD bottles and other apparatus must be kept clean. Bottles should be cleaned with sulfuric acid dichromate solution and rinsed well with DI water.

Dilution Water

Measure sufficient DI water (usually 6 L) with a graduated cylinder and add sufficient BOD nutrient buffer pillows (e.g., I Hach buffer pillow is for 6 L of water). Shake the jar very well to dissolve the contents of the pillows (capsul).

Sample Size

When the sample is not known, the appropriate strength of the sample size can be determined according to the following procedure. Assume that the BOD of the sample is equal to 100 and the depletion is half the original DO. For example, if the $DO_1 = 8$ and $DO_2 = 4$, the **first sample size** (SS_1) is

$$SS_1 = \left(8 - \frac{8}{2}\right) \times \frac{300}{100} = 12\,\text{ml}$$

(Note: The BOD bottles hold 300 ml of water.) To find the other two sizes, the following conditions must be met. First, the sample must deplete at least 2.0 mg/L of the original DO content of the dilution water. Based on this, the **second sample size** (SS_2) is

$$SS_2 = 2.0 \times \frac{300}{100} = 6\,\text{ml}$$

Second, the sample must leave 1.5 mg/L of DO after 5 days of incubation. Therefore, the **third sample size** (SS_3) will be

$$SS_3 = (8 - 1.5) \times \frac{300}{100} = 19.5\,\text{ml}$$

The three sample sizes to be used in this example are 12, 6, and 19.5 ml. When the range of the BOD is known, you can use the following formula to find the appropriate dilution sizes:

$$\left(\text{DO}_{\text{Blank}} - \frac{\text{DO}_{\text{Blank}}}{2}\right) \times \frac{300}{\text{BOD}}$$

For example, if the expected BOD in the sample is between 9 and 158 and the DO of blank is equal to 8, the lowest sample size is

$$\left(8 - \frac{8}{2}\right) \times \frac{300}{9} = 133.3\,\text{ml}$$

and the highest sample size is

$$\left(8 - \frac{8}{2}\right) \times \frac{300}{158} = 7.6\,\text{ml}$$

Sample Preparation and DO Measurement

Fill one BOD bottle only with dilution water and use it as the *blank*. On the blank sample, the DO uptake (initial DO–DO after five days of incubation) should not be more than 0.2 and preferably not more than 0.1 mg/L. A blank with a DO uptake above this indicates contamination in the bottle or the dilution water.

Fill another bottle about half full with dilution water from the side of the bottle in order not to mix air with the water. Then, with a pipet and with the tip below the level of the water, add the *proper amount of the sample*. Completely fill the bottle with the dilution water, place its stopper, and shake well (see the Note).

Measure the initial DO of the *blank* and *samples* as follows:

1. Insert the funnel of the BOD electrode (here, refers to Orion BOD electrode) into the bottle and immerse the electrode into the funnel.
2. Place the bottle on a magnetic stirrer and stir gently.
3. Release the meter (refers to Orion pH meter) from standby by pressing any key and set it to pH mode.
4. Turn the electrode's switch to the H_2O position, allow the reading to stabilize, and record the reading (to the nearest 0.1 ppm).
5. Turn the electrode's switch to the off position.
6. Rinse the funnel and electrode with DI water between samples, dry the electrode, and gently blot the membrane dry.
7. Place the funnel and the electrode in the storage bottle (the air calibration bottle) and read the DO content.
8. Place the bottles in the incubator at 20°C.
9. After 5 days of incubation, take out the *blank* and the *samples*, and run the DO as explained in steps 1 through 7.

NOTE

Always arrange the BOD sample bottles for reading in the order of increasing numbers so that the lowest number is the blank.

Testing the BOD with Seeded Samples

To test a seeded sample, proceed as follows:

1. Place the contents of one seed capsule (BOD seed, available from a lab supplier e.g., fischer polyseed) into a 500 ml of dilution water. Aerate and stir the solution for 1 hour. For the best result, the seed solution should be used within 6 hours of dilution.
2. Prepare three BOD bottles. Add 10, 15, and 20 ml seed solution, respectively, to each bottle. Fill them to the appropriate level with dilution water so that the insertion of the stopper will displace all air, leaving no bubbles in the bottle.
3. Fill one BOD bottle with dilution water without adding any seed to it.
4. Determine the initial DO of each prepared bottle immediately after preparation. Incubate the bottles at 20°C for 5 days. After 5 days of incubation, determine the DO of each bottle.

Calculation

When seed is not used, calculate BOD (in mg/L) according to the following equation:

$$BOD = \frac{DO_1 - DO_2}{F} \text{ mg/L}$$

DO_1	Initial DO of sample
DO_2	DO after 5 days incubation
F	Dilution factor (ml sample × 100/300 or ml sample/3)

When **using seed**, BOD is calculated according to the following formula:

$$\text{BOD} = (\text{DO}_1 - \text{DO}_2) - (B_1 - B_2) \times \frac{V}{F}$$

B_1	Initial DO of seed control
B_2	DO of seed control after 5 days
V	ml sample in seed control (if the amount of seed solution in the seed control and sample bottles is the same, V equals 1)

Standardization of DO Electrode

To standardize Orion BOD electrode (according to its instruction), proceed as follows:

1. Place approximately 1.5 cm of DI water in a BOD bottle. Insert the funnel of the electrode into the bottle after wiping the probe carefully with a tissue. The probe must not touch the water while calibrating. Use always this bottle for holding and storing the electrode between tests.
2. With the electrode's switch in the Off position, put the meter on pH mode.

3. Press the Hold key. Turn the Hold feature off.
4. Press the Measure key. Press the Set-Temp key. Enter 25.0 at the prompt and press the Yes key.
5. Press the Calibrate key. Enter 1 at the prompt and press the Yes key.
6. Enter 7.0 and press the Yes key.
7. At the slope prompt enter 100.0 and press the Yes key. Wait until the meter automatically returns to the measure mode.
8. Turn the mode the electrode's switch to BT CK. Good battery operation is indicated by a reading of 13.0 or greater on the meter.
9. Turn the electrode's switch to Zero. Use the electrode's zero calibration control to set the meter to 0.
10. Turn the electrode's mode switch to the Air position by using the air calibration knob and set the barometric pressure of the area (see the Notes).
11. Insert the electrode into the funnel; switch the electrode selector to H_2O mode.
12. Place the bottle on the magnetic stirrer and let it stir gently at a constant rate. When the reading has stabilized, record the reading.
13. Rinse the funnel and electrode with DI water, dry the electrode, and gently blot the membrane dry.

NOTES

- **Barometric pressure = 760 × elevation correction factor = 760 × 0.982 = 746 mm mercury. The elevation correction factor can be found in a table provided in the Instruction Manual for the oxygen electrode. For example, if the city is 150 m above sea level, the elevation correction factor is 0.982.**
- **The value of COD is higher than BOD by about 2 to 1. The ratio between the two values depends on the type of the sample.**

SOLIDS IN WASTEWATER

The wastewater of a beet-sugar factory contains high amounts of **suspended** and **dissolved solids**.

Total Suspended Solids (*TSS*)

To test a wastewater sample for *TSS*, filter 50 ml of the sample through filter paper with medium porosity (almost 10 µm pore size) and record the following:

1. Mass of filter and residue (after drying at 105°C for 45 minutes), in grams
2. Mass of empty filter, in grams
3. Subtract recorded item 2 above from item 1 to calculate *TSS* in mg/L (ppm)

$$TSS = \text{Item } 3 \times \frac{1000000}{\text{ml}_{\text{Sample}}}$$

For example, for 50 ml sample, the formula is

$$TSS = \text{Item } 3 \times 20000$$

Collect the filtrate for use in the next test.

Total Dissolved Solids (*TDS*)

Evaporate the filtrate from the preceding test in a weighed dish (crucible). Dry the dish of filtrate at 105°C, and record the following:

4. Mass of dish and dry residue, in grams
5. Mass of empty dish, in grams
6. Subtract item 4 from item 5 to calculate *TDS* in mg/L (ppm).

$$TDS = \text{Item } 6 \times \frac{1000000}{\text{ml}_{\text{Sample}}}$$

For a 50 ml sample, the formula is

$$TDS = \text{Item } 6 \times 20000$$

Total Solids (*TS*)

$$TS = TSS - TDS$$

or evaporate 50 ml of well-mixed sample in a crucible at 105°C to dryness. The increase in weight over that of the empty dish represents the total solids.

Volatile Suspended Solids (*VSS*)

To calculate **volatile suspended solids** (organic suspended solids), proceed to record as follows:

7. Mass of empty dish (crucible)
8. Mass of empty filter item 2
9. Add item 7 and item 8
10. Add items 1 and 7
11. Dish and residue (after ignition at 550°C for 15 minutes)
12. Subtract item 10 from item 11 to calculate *VSS* in mg/L (ppm)

$$VSS = \text{Item } 12 \times \frac{1000000}{\text{ml}_{\text{Sample}}}$$

For a 50 ml sample,

$$VSS = \text{Item } 12 \times 20000$$

Fixed Suspended Solids (*FSS*)

To calculate **fixed suspended solids** (inorganic suspended solids), proceed as follows:

13. Same as item 11
14. Same as item 9
15. Subtract item 13 from item 14 to calculate *FSS* in mg/L (ppm)

$$FSS = \text{Item } 15 \times \frac{1000000}{\text{ml}_{\text{Sample}}}$$

For a 50 ml sample,

$$FSS = \text{Item } 15 \times 20000$$

NOTE

mg/L = ppm, and 10000 mg/L = 1% solution

NITROGEN IN WASTEWATER

The **total nitrogen test** determines the quantity of nitrogen or ammonia in the water sample. The nitrogen can be determined by an **electrode method**. Unlike Kjeldahl method, the **electrode method** does not need distillation. It is determined by the potentiometric method. The range of the test is 0.01 to 100 mg/L with higher levels requiring dilution. To check accuracy, a nitrogen standard solution is used. The result gives total nitrogen (N) in ppm. To calculate ammonia (NH_3), the result is multiplied by 1.21.

Equipment

- A pH meter with mV readability, such as Orion 720 A or equivalent
- Ammonia electrode, such as Orion
- Pipettes, 1, 10, and 50 ml capacity
- Magnetic stirrer
- 100-ml, 500-ml, and 1000-ml flasks
- 300-ml beaker

Reagents

Lab pure water: If ammonia free is unavailable, use distilled water and add 0.1 ml H_2SO_4 per liter of water. Prepare fresh each day.

Sodium hydroxide 5 *N*: Dissolve 20 g of NaOH in about 80 ml of freshly boiled and cooled DI water in a 100-ml flask; dilute to the mark.

Nitrogen stock solution (1000 mg nitrogen per L or 1000 ppm): Dissolve 1.91 g of NH_4Cl in 500 mL DI water (equivalent mass of NH_4Cl = 53.5, equivalent mass of *N* = 14.0, 53.5/14.0 = 3.82 g/L, or 1.91 g/500 ml) that has been dried at 105°C for 1 to 2 hours and cooled in a desicator.

Standard Solutions

To prepare standard solution, proceed as follows:

1. **Nitrogen standard solution (100 ppm)**: Pipet 10 ml of stock solution into a 100-ml flask and dilute to volume with distilled water.
2. **Nitrogen standard solution (1 ppm)**: Pipet 1.0 ml of preceding solution into a 100-ml flask and dilute to volume.

Standardization of the Electrode

To standardize Orion ammonia electrode (according to its instruction), proceed as follows:

1. Place the ammonia electrode in a beaker of distilled water for a few minutes, and then wipe and dry it.
2. Pour the first standard solution (1 ppm) into a 150-ml beaker with a stirring bar, and add 2.0 ml (for 100 ml of sample) sodium hydroxide while stirring the solution. Sodium hydroxide is used for adjusting the pH to the operating range of the electrode.
3. Take the electrode from the distilled water, wipe it dry, put it into the lowest standard solution (1.0 ppm), and press Calibrate. The date and time of the last calibration is displayed. Press 2, then press Yes to select the two-stage calibration.
4. When the Ready Enter Value is displayed, indicating that the electrode is stable, enter the value of the first standard (1.0 ppm) and press Yes. After three seconds, the meter automatically switches to Standard 2. If the reading for the 1.0 ppm standard is between 54 and 60 mV, the probe is operating properly. If not, the membrane probably needs to be replaced.
5. Pour 100 ml of the second standard (100 ppm) into another 150-ml beaker, insert the electrode, and add 2.0 ml of sodium hydroxide while stirring.
6. Remove the probe from the first standard, and rinse and blot it dry.
7. When the Ready Enter Value is displayed, enter the value of the second standard (100 ppm) and press Yes.
8. The electrode slope is calculated and then the actual value is displayed for five seconds. The meter then automatically proceeds to the Measure mode.
9. Remove the electrode from the standard. Rinse with distilled water and blot dry. Place the electrode into the sample. When Ready is displayed, indicating that the reading has stabilized (it takes 2 to 3 minutes), record the result.

NOTES

- **Between measurements or for overnight storage, keep the electrode tip immersed in 1000 ppm standard *without the added NaOH solution*. For storage over one week or if the electrode is stored indefinitely, disassemble completely and rinse all parts of the electrode with distilled water. Dry and reassemble it without filling solution or membrane.**
- **A membrane will last from one week to several months depending on usage. Membrane failure is characterized by a shift in electrode potential, drift, and poor response. Membrane failure may be apparent on visual inspection as dark spots or discoloration on the membrane.**

Sample Analysis

To test a sample for ammonia using Orion ammonia electrode and pH meter, proceed as follows:

1. Measure 100 ml of the sample into a 150-ml beaker with a stirring bar, and add 2 ml of 5 *N* sodium hydroxide solution. Samples should be measured at room temperature.
2. Put the beaker on the magnetic stirrer and stir thoroughly at a constant rate.
3. Rinse the electrode with distilled water and place into sample. Press any button to release the Orion meter from standby.
4. Press Mode key until Con is displayed. When the reading has stabilized, record the result.

CHAPTER
11

SECTION 10
LABORATORY SAFETY AND FIRST AID

SUBSECTIONS

- Chemical Hazards
- Chemical Hazard Labels
- First Aid

When you work in a lab, you are responsible not only for your own safety but also for the safety of all other in the lab, so **lab safety** should be a major concern for everyone. The lab is considered a hazardous environment, thus, safety precautions must be carefully followed when testing samples. Before working in the lab, all personnel must know the following lab safety rules:

1. Do not smoke in the lab
2. Do not eat or drink in the lab
3. Do not use the lab glassware to drink
4. Do not pipet a chemical solution by mouth
5. Do not wear inappropriate clothing in the lab
6. Do not conduct unauthorized experiments in the lab
7. Do not leave jackets or other personal items on the lab bench
8. Wear safety glasses when you work with acid and base
9. Ask a responsible person when you are in doubt
10. Handle chemicals and glassware cautiously
11. Ask for assistance when you are injured
12. Avoid direct contact with chemicals
13. Clean chemical spills immediately
14. Keep the lab environment clean
15. If your hair is long, tie it back when working with a burner
16. Know where the **material safety data sheets** (MSDS) are kept
17. Label all poisonous solutions *POISON* and handle them with caution
18. Mercury is POISONOUS and can be absorbed by skin and inhalation

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19. Work with acid and base solutions cautiously and under the fume hood
20. If a thermometer containing **mercury** breaks, seek assistance in cleaning
21. If you come in contact with chemicals, wash the affected part with water
22. If a chemical splashes into your eyes, go to the eyewash station immediately
23. Add acid to water slowly, NEVER ADD WATER TO ACID (this causes a violent reaction)

The following precautions must be followed by the person in charge of the lab:

- Label all chemical bottles
- Store toxic chemicals in a ventilated area
- File all **material safety data sheets** (MSDS) because the MSDS sheet of each chemical contains all precautions and dangers associated with that chemical. (By law, all chemical suppliers must provide the users with MSDS sheets.)
- Store all flammables in the flammable storage cabinet
- Prepare a list of lab chemicals on hand and update the list as new chemicals are received

CHEMICAL HAZARDS

Chemical hazards are harmful to human health to some degree and some of them are toxic even in a small amount. In the United States, the producers of chemicals must provide the buyers a **Material Safety Data Sheet** (MSDS) for their chemicals. Buyers must keep the MSDS files in the facility for their employees access. The employers must also train their workers on chemical hazards. Chemical hazards are divided into the following four groups:

- **Corrosive hazards**: Chemicals with a pH of 3 or less, or a pH of 12 or more.
- **Ignitable hazards**: Chemicals with the ability of causing a fire. An **ignitable liquid** has a **flash point** (the lowest temperature at which an ignitable substance react with air) less than 60°C. The flash point is slightly lower than the **fire point**.
- **Reactive hazards**: Chemicals with one of the following characteristics:

 Reacts violently with water (generates toxic vapor or fume)
 Unstable and readily undergoes violent chemical reaction
 Capable of exploding at ordinary room temperature
 A cyanide or sulfide containing substance

- **Toxic hazards**: Chemicals with the ability to cause harm to human health when inhaled, swallowed, or absorbed through the skin. Even some household products (e.g., laundry detergent) contain toxic chemicals. **Lethal dose** (LD) is the amount of a toxic substance that causes the death of humans. For example, minimum LD of fluoride is 5 mg/kg (i.e., 5 mg for each kg bodyweight). **Toxicology** is the study of the effects of toxic substances on humans.

CHEMICAL HAZARD LABELS

Chemical hazard labels (pictographs) are applied to containers of hazardous (dangerous) chemicals to indicate the specific risk. As Figure 11.24 indicates, each label contains four color-coded boxes for type of hazards; each box has a number indicating severity.

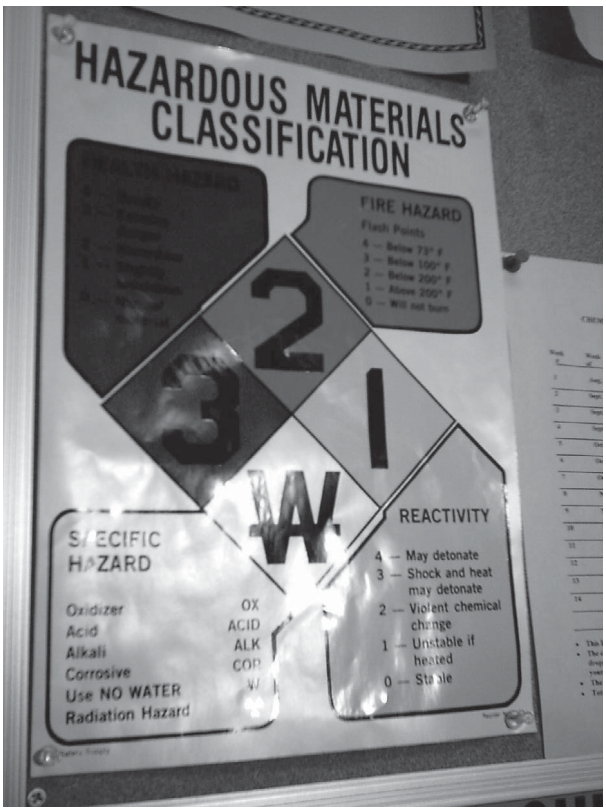


Figure 11.24 Hazards classification (Source: the National Fire Protection Asscіation, NFPA)

Types	Severity
Blue: Health	0: No hazard
Red: Flammability	1: Slight hazard
Yellow: Instability	2: Moderate hazard
White: For special hazard information	3: Severe hazard

FIRST AID

First aid is the immediate-temporary-life-saving treatment provided to an injured or sick person until medical help can be given. First aid can be provided by a non-doctor with minimal training and with minimal equipment. The following are some first-aid recommendations:

- **Chemical injury**: When someone is injured by an **acid** or a **base**, the person should be moved to the washing shower station of the laboratory (see Figure 11.25) and the affecting areas should be washed gently with water.



Figure 11.25 Shower station

- **Eye injury**: In the case of chemical **eye injury** (by acid, base, or irritant gas), the following procedure is helpful:
 1. Immediately wash the eye with water.
 2. Apply an eye wash solution.
 3. Apply an eye ointment.
 4. Send the injured person to a medical facility if necessary.

- **An object in the eye**: In the case of an object in the eye, remove the object gently with the corner of a handkerchief moistened in water. If the object remains under the upper lid, pull the upper eyelid over the lower eyelid and repeat this action several times.
- **Wounds**: In the case of a wound, a bandage applied over the wound is usually all that is required. If bleeding persists and the wound is on the arm, leg, or foot, apply a tourniquet (a piece of rubber tubing makes a good tourniquet) 10 cm (4 in.) below the armpit or the **groin** (the fold on both sides of the body between the abdomen and the upper thigh) to stop the flow of blood. If the bleeding is in a position where a tourniquet cannot be applied,

NOTES

- **A tourniquet should be released every 20 minutes and reapplied if bleeding resumes. In any case, an injured person who continues to bleed must be transferred to a hospital as soon as possible.**
- **The information given in this section is general and usually each country has its own official guidelines on how the first aid should be provided.**

bandage the wound firmly with a first-aid dressing and transfer the injured person to the **hospital** immediately.

In the case of a **burn** caused by an **acid**, wash the area first with cold water and then with diluted sodium bicarbonate solution, apply gauze, and bandage lightly. If the burn is caused by an **alkali**, wash the area first with cold water and then with a 2% acetic acid solution, apply gauze, and bandage it lightly. If the burn is serious, the person should be transferred to the hospital immediately.

In the case of **electrical shock**, immediately turn off the current. If this is impossible, remove the patient by physical means, taking every precaution to insulate yourself from contact by wearing thick gloves and standing on a nonconducting material. If the patient is not breathing, call an ambulance, start **artificial respiration** and cardiac massage until the ambulance arrives.

Artificial Respiration

Artificial respiration is the flowing of air in and out of the lungs of a person who is not breathing adequately, but his/her heart is still beating. In emergency situations, mouth-to-mouth or mouth-to-nose respiration is effective. It consists of the following steps:

1. Place the patient on his or her back, with the head tilted backward and the chin pointing upward so that the tongue does not block the airway.
2. Keep the patient's mouth open with one hand and the nose closed with the other hand.
3. Take a deep breath and blow into the patient's mouth or nose.
4. Repeat the inflation until natural breathing resumes or until professional help arrives.

If the person is unconscious, not breathing, and appears to have no pulse, apply both artificial respiration and cardiac massage.

Cardiac Massage

Cardiac massage consists of the following steps:

1. Place the patient face up on a firm surface and have someone else give the patient respiration.
2. Sit beside the person and apply the palm of one hand over the bottom of the breastbone. Bring the palm of the other hand on top of the first, and then press down and up about 60 times a minute, applying your body weight evenly.

END-OF-THE-CHAPTER NOTE

- **In Chapter 11, you (mainly refers to sugar chemists) have learned about the most important analytical instruments used in sugar laboratories. Furthermore, you have learned about some subjects on the preparation of a factory's reports of operations, methods of analysis, and preparation of the laboratory reagents. Generally, laboratory techniques require a deep knowledge of the basics of science, which are given in the next chapter. Remember there is some laboratory-related information in the Appendix as well.**

CHAPTER 12

BASICS OF SCIENCE RELATED TO SUGAR TECHNOLOGY

SECTIONS

1. Basics of Chemistry
2. Basics of Mathematics and Statistics

ABOUT THE CHAPTER The goal of **Chapter 12** is threefold:

- To give the basics of science related to sugar technology, which relates in one direction with chemistry and in another with mathematics.
- To give the basics of statistics because every person involved with math and data needs to know statistics as a systematic and efficient procedure for describing data.
- To give practical examples for nearly all equations to improve the reader's understanding.

Individuals familiar with the basics of science can use this chapter as a **reference**. Those unfamiliar with the basics can use it as a **science tutorial**. As you will notice, learning basics of science is not difficult–it just takes time.

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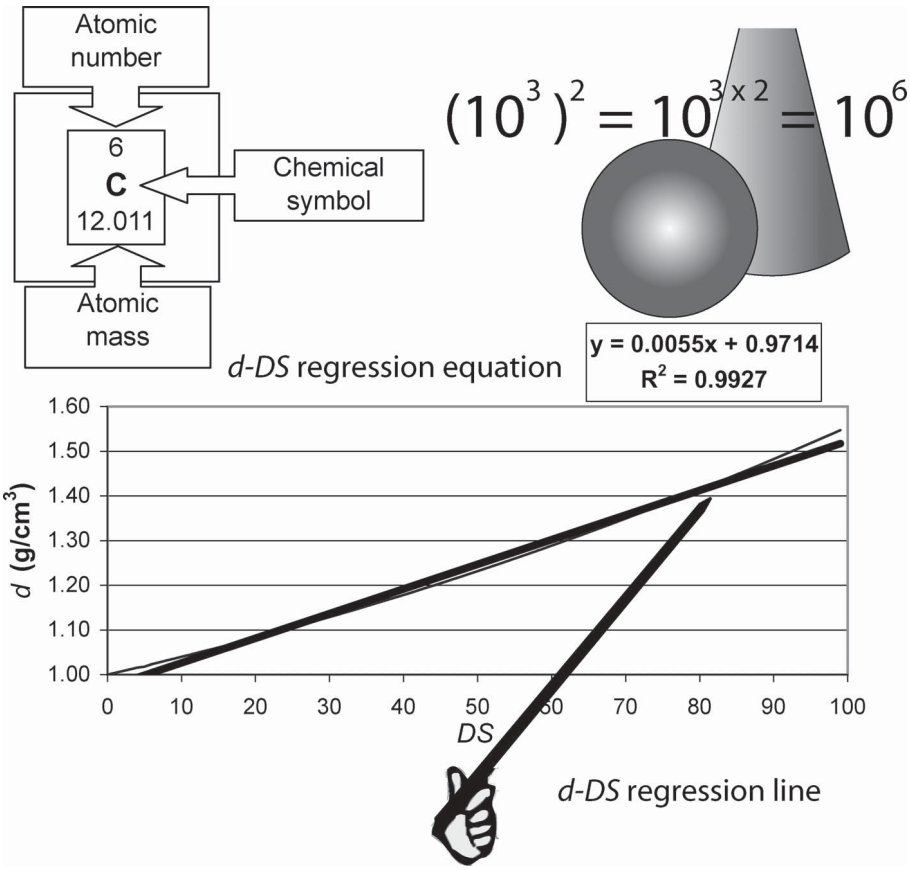


Figure 12.1 Knowing the basics of chemistry, mathematics, and statistics can improve our understanding of sugar technology

CHAPTER
12

SECTION 1
BASICS OF CHEMISTRY

SUBSECTION

- Mass, Weight, Density, and Specific Density
- Periodic Table of Elements
- Atom and Molecule
- Atomic and Molecular Mass
- Mole, Molar Mass, and Mole Fraction
- Equivalent Mass and Number of Equivalent
- Concentration of Solutions
- Molality
- Molarity
- Normality
- Chemical Formula and Chemical Equation
- Acid and Base
- Standard Solution Preparation
- Titration
- Meniscus
- Osmosis and Reverse Osmosis

ABOUT THIS SECTION **This section** provides the **basics of chemistry** to the level required by a sugar chemist or a sugar technologist. Sugar chemists need the information provided here even more than sugar technologists because laboratory work, such as the preparation of **laboratory reagents** and **standard solutions**, requires good knowledge of chemistry topics such as mole, equivalent mass, molarity, normality, etc. These subjects and more are provided in this section.

History: Antoine Lavoisier (1743–1794), a French chemist established the foundation of modern chemistry. He was the first to represent the notion of **element** and **compound**.

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A **compound** is composed of two or more elements (the simplest types of matter, e.g., calcium) that are chemically **bond** (linked). Water (H_2O), sucrose ($C_{12}H_{22}O_{11}$), salt (NaCl), calcium carbonate ($CaCO_3$), and ammonia (NH_3) are examples of compounds. Millions of compounds exist, but all are divided into two main groups.

Chemistry is the study of the properties of substances (matter) and their chemical reactions. Substances are

- **Element**: Any substance made of one kind of atom. For example, hydrogen (H) is an element because it is composed of only hydrogen atoms.
- **Compound**: Any substance made of two or more elements. Compounds are organic and inorganic. **Organic compounds** are made of carbon atoms bonded (connected) to each other and to atoms of other elements. For example, sucrose ($C_{12}H_{22}O_{11}$) consists of carbon atoms bonded to oxygen and hydrogen. **Inorganic compounds** do not contain carbon. For example, sodium hydroxide (NaOH) and sulfuric acid (H_2SO_4) are inorganic compounds. A few **carbon-containing** compounds such as calcium carbonate ($CaCO_3$) and carbon dioxide (CO_2) are inorganic compounds because they do not contain C–H bonds.

Chemistry is divided into two major branches:

- **Organic chemistry**: Deals with carbon (C) containing substances
- **Inorganic chemistry**: Deals with the rest of substances

Although **sugar** (sucrose) is an organic compound, but sugar technology mostly involves with inorganic chemistry and to a lesser extent with organic chemistry.

MASS, WEIGHT, DENSITY, AND SPECIFIC DENSITY

Mass and **weight** are used interchangeably. Strictly speaking, this is incorrect because

- **Mass** is the measure of the quantity of a substance, but **weight** is the measure of gravitational force (F_G or force of Earth's gravity) applied to an object.
- **Mass** is not dependent on our location in the Universe, but **weight** is. For example, our mass is the same on the Moon and on the Earth, but our weights would be much less (by one-sixth) on the Moon than on the Earth because the F_G on the Moon is one-sixth that of the Earth.
- The unit of **mass** is kilogram (kg), but the unit of **force** is Newton ($1\,N = 1\,kg.m/s^2$)

When we weigh a sample on a balance in the lab, we are comparing the mass of that sample to a standard mass. Therefore, even though we say that we weighted the sample, we actually determined the mass of the sample. So, the correct way to express the action of weighing is: *the **mass** of the sample was measured on the balance and it was found to be so and so.*

NOTES

- **The term mass is used in this book to the extent that it does not create any confusion. For example, in expressing percentages, percent by mass (% m/m) is used instead of percent by weight (% w/w) or molecular mass is used instead of molecular weight.**
- **Einstein equation, $E = mc^2$, expresses the relationship between mass (m) and energy (E). c is the speed of light. This is the key to understanding how 1.0 g of matter (if totally converted to energy) contains enough energy to cover the electricity of a house for about 1000 years.**

Density (d) is the mass per unit volume. Mass may be in kilograms (kg) or pounds (lb), and volume (V) may be in cubic meters (m^3) or cubic feet (ft^3). Mathematically, density is given as

$$d = \frac{\text{Mass}}{V} \tag{12.1}$$

Specific density (specific gravity) is the mass of a substance divided by the mass of an equal volume of water at a specified temperature. The standard substance for liquids is **water** and for gases is **air**. Specific density has no units because the units in the numerator and the denominator cancel. Because the density of water is numerically equal to its specific density, the specific density of a substance is numerically equal to its density, so density and the specific density are usually used interchangeably. The specific density (d_{Sp}) is given by

$$d_{Sp} = \frac{d_{\text{Sample}}}{d_{\text{Water}}} \tag{12.2}$$

For example, the density of a sucrose (sugar) crystal (see Notes) is 1586 kg/m^3, so its d_{Sp} is

$$d_{Sp} = \frac{1586\,(kg/m^3)}{1.0\,(kg/m^3)} = 1586$$

EXAMPLE 1

Given:

Mass of a sample	1.90 g
Volume of the sample	1.65 ml

Calculate:
Density (d) of the sample

$$d = \frac{\text{Mass}}{V} = \frac{1.90}{1.65} = 1.15\,g/ml$$

NOTES

- **Bulk density of sugar (sucrose) is 840 to 880 kg/m^3 depending on the size of crystals *(the finer the crystals, the larger the bulk density of sugar).***
- **The density of liquid of a substance is larger than that of its gas because the molecules in liquids are closer than the molecules in gases.**
- **In determining density, the mass of a substance can be obtained using a weighing balance, but the volume may or may not be easy to obtain. The volumes of some objects are given in Table A.18 (Useful Formulas from Geometry) in the Appendix.**
- **The density of irregular-shaped solids can be determined with the help of a graduated cylinder, as follows:**
 1. **Determine the mass of the object.**
 2. **Partially fill a graduated cylinder with a liquid and record the volume.**
 3. **Submerge the object in the water, and record the volume again.**
 4. **The difference in the measured volume is the volume of the object.**
 5. **Divide the mass of the object by the volume.**

The density of liquids can be determined by pycnometer (density bottle), as follows:

1. **Determine the mass of the empty density bottle (M_1).**
2. **Fill the bottle to the top of the capillary with the sample.**
3. **Determine the mass of the filled bottle (M_2).**
4. **Obtain the mass of the sample by subtraction ($M_2 - M_1$).**
5. **Rinse bottle with deionized (DI) water, fill it again at 20°C, and weigh it (M_3).**
6. **The mass of the water is equal to $M_3 - M_1$.**
7. **Divide the mass of the sample by the mass of water to determine the density.**

PERIODIC TABLE OF ELEMENTS

The **periodic table** (given inside the back cover) is an organized table containing the symbols of all the elements with their **atomic numbers** and **atomic masses** (atomic weights). D. I. Mendeleev (1834–1907), a Russian chemist, arranged the periodic table in 1869, and its revised version is still used today. About 118 elements are known; of these, about 90 are found in nature, and the remainder has been created in the laboratory. Scientists even gave names and symbols to a few elements that have not been observed.

Each **element** (the simplest type of substance) has a name and a chemical symbol. The chemical symbol is a one-letter or two-letter abbreviation for each element. For example, K and Mg are chemical symbols for potassium and magnesium, respectively.

The periodic table helps us predict properties of elements because it is based on similarties in the chemical and physical properties of elements. It is organized according to **atomic number** (the number of protons) of elements. Elements are organized by increasing atmic number into **groups** (columns) and **periods** (rows). The table has 18 groups and 7 periods. Elements with similar properties are placed into a group. For historical reasons, some groups are known by their family names, such as the **alkali metals** Mg, Ca, and Ba (because they react with oxygen to form alkalis [bases]), and the **alkaline earth metals** Na and K (because their presence makes soil alkaline [basic]).

All the elements on the left side and in the middle up to the stair step (except for hydrogen) are metals. Metals share many similarities, such as high electrical and heat conductivity. All metals (except mercury) are solids at **room temperature** (around 20°C). Many metals mix easily when melted. Such mixtures are called an **alloy** (mixture of metals). For example, **stainless steel** is an alloy consisting of iron, chromium, and nickel. The best-known nonmetals are hydrogen (H), oxygen (O), helium (He), carbon (C), nitrogen (N), fluorine (F), chlorine (Cl), bromine (Br), iodine (I) silicon (Si), phosphorus (P), sulfur (S), and argon (Ar).

The **atomic number** is the total number of electrons in an element. The atomic number gives also the number of protons because elements have the same number of electrons and protons. The **atomic mass** is the total number of protons and neutrons in an atom. In each box of the table, the number above each symbol is the **atomic number** and the number below the symbol is the **atomic mass** (more on the subject of atomic mass later). For example, carbon (see Figure 12.2) has atomic number 6 and atomic mass 12.011

NOTE

Chemistry related to sugar technology requires familiarity with 15 to 20 elements. A list of common elements used in sugar technology and sugar lab practices, along with their symbols and atomic masses, is given inside the back cover of this book, next to the Periodic Table.

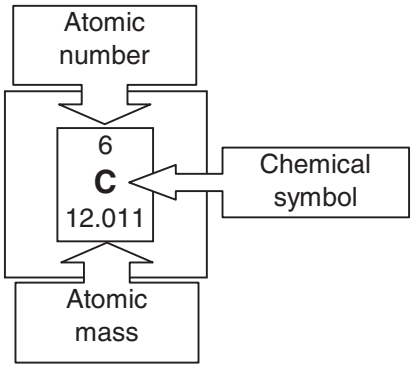


Figure 12.2 Periodic Table segment

ATOM AND MOLECULE

An **atom** is the smallest particle of an element. Atom cannot be decomposed by ordinary chemical means. Every atom consists of the following:

- **Nucleus:** Made of **protons**, which have positive charge and **neutrons** with no charge, so nucleus carries positive charge.
- **One or more electrons:** Are in rapid motion around the nucleus, forming a negative electrical charge (opposite to that of a proton).

The atom is electrically **neutral** because the positive charge of protons neutralizes the negative charge of electrons (e^-). The total mass of protons and neutrons in each element equals the mass of the atom because the mass of the electron is negligible. In other words, the nucleus carries almost all the mass of the atom (99.9%). The lightest atom is hydrogen (H), which has a mass of 1.67×10^{-24} g. A carbon (C) atom is 12 times heavier and has a mass of 20.04×10^{-24} g.

A **molecule** consists of different atoms joined together chemically. It is the smallest unit of a substance that can exist by itself and keep all the properties of that substance. Most substances exist as molecules, not as atoms. The molecules of some substances consist of very few atoms, such as water (H_2O), which has a molecular mass of 18 g. But some molecules are large, such as polymers, which are made from thousands of repeating units with a high molecular mass.

Atoms or molecules are bonded (connected) to form compounds in three major ways:

- **Ionic bonding**: Ionic bonds occur when atoms combine by losing or gaining electrons. For example, when sodium (Na) combines with chlorine (Cl) to form NaCl, the Na atom loses one of its electrons and the Cl atom gains one electron. As a result, the Na atom becomes a **cation** (positive ion) and the Cl atom becomes an **anion** (negative ion).
- **Covalent bonding**: Covalent bonds occur when two atoms combine by sharing electrons. The shared pair electron in the molecule is called the **covalent bond**. For example, in a hydrogen molecule (H_2), two electrons (one from each H atom) are shared between H atoms. Similarly, in an oxygen molecule (O_2), four electrons (two from each O atom) are shared between O atoms. The **oxygen** (glycosidic) **bond** between glucose and fructose molecules in a sucrose molecule is also a covalent bond (see Section 4 in Chapter 1).

- **Hydrogen bonding**: Hydrogen bonds occur between molecules that contain H atoms. For example, the bonds between neighboring water molecules are **hydrogen bonds** as well as the bonds between sucrose molecules. The hydrogen bonding is responsible for the high **boiling point** (*BP*) and **melting point** (*MP*) of some substances that contain hydrogen bonds.

NOTES

- **To better understand the chemistry, we must elevate our imagination because we cannot see atoms, molecules, and their behavior in different reactions.**
- **Most elements have one atom in their molecules, such as sodium (Na) or potassium (K). But there are seven elements, known as diatomic elements, with two atoms in their molecules. In diatomic molecules, two atoms of the same element are joined by a covalent bond to from a molecular element (e.g., molecular hydrogen, H_2). The diatomic elements are**
 - **Hydrogen (H)**
 - **Oxygen (O)**
 - **Fluorine (F)**
 - **Chlorine (Cl)**
 - **Bromine (Br)**
 - **Iodine (I)**
 - **Nitrogen (N)**
- **Atoms interact with one another electronically (based on electron). Therefore, two situations are possible:**
- **Oxidation: The loss of an electron (or electrons) by an element or a compound. The element or compound that loses the electron (e^-) is oxidized.**
- **Reduction: The gain of an electron (or electrons) by an element or a compound. The element or compound that receives the electron is reduced.**
- **Reducing agents are substances that reduce another substance by giving an electron, so become oxidized during an oxidation-reduction reaction.**
- **Oxidizing agents are substances that oxidize another substances by accepting an electron, so become reduced during an oxidation-reduction reaction. For example, oxygen in the air oxidizes an automobile's metals.**

ATOMIC AND MOLECULAR MASS

Atomic mass (atomic weight) is the total mass of an atom. This is the sum of the masses of nucleus (protons and neutrons) and electrons (its mass is negligible). Because the mass of an atom is so tiny, scientists developed a unit for atomic mass, known as the **atomic mass units** (AMU). AMU is based on C–12 (read carbon 12). C–12 has a mass of exactly 12 AMU. The masses of all other atoms are given relative to C–12. For example, the atomic mass of magnesium (Mg) is 24 AMU, which means Mg is two times heavier (more massive) than carbon (C). Therefore, when we weigh twice as much Mg as C, we obtain the same number of atoms. Now we are in a position to connect the AMU with the number of atoms in an element and express the atomic masses in **grams** ($1\,\text{AMU} = 1.66 \times 10^{-24}\,\text{g}$). There are 6.02×10^{23} atoms in 12 g of C or 24 g of Mg. For simplicity, in chemistry the atomic masses and molecular masses are given in grams.

Molecular mass (*MM*, also called **molecular weight**, or **formula mass**) is the sum of the atomic masses of all atoms in a molecule. In other words, *MM* (or *MW*) is the mass of one molecule of a substance in grams.

NOTE

Elements usually consist of a mixture of isotopes (two or more atoms with the same number of protons but a different number of neutrons). If an element has few isotopes, the weighted average (average giving points to the priorities) mass of its isotopes is used as its atomic mass. For example, carbon exists in nature as two isotopes: 98.9% in the form of ^{12}C with an atomic mass of 12.0000 g and 1.1% in the form of ^{13}C with atomic mass of 13.0034 g. Thus, the atomic mass of carbon is

$$12.0000 \times 0.989 + 13.0034 \times 0.011 = 11.868 + 0.143 = 12.01\,g$$

EXAMPLE 2

What is the molecular mass (*MM*) of the following compounds?
Sucrose (sugar, $C_{12}H_{22}O_{11}$)
Calcium oxalate (CaC_2O_4)

Sucrose ($C_{12}H_{22}O_{11}$) consists of the elements of C, H, and O, and its *MM* is

$$(12 \times 12.01) + (22 \times 1.008) + (11 \times 16.0) = 342.3\,g$$

CaC_2O_4 consists of the elements of Ca, C, and O, and its *MM* is

$$(1 \times 40.0) + (2 \times 12.01) + (4 \times 16.0) = 128.0\,g$$

We are now ready to learn about the conversion of a substance's quantity from **mass** to **mole**. In other words, we want to find how many moles are in so many grams of a substance. **Mole** is used very often in chemistry, so it is defined next.

MOLE, MOLAR MASS, AND MOLE FRACTION

Mole (*n*) is the mass (in grams) of a substance (element or compound) equal to its atomic mass (in case of an element) or molecular mass (in case of a compound). In other words, if you weigh a quantity of a substance equal to its atomic or molecular mass, you also have 1 mole of that substance. Scientifically, mole is the mass (in grams) of 6.02×10^{23} particles (atoms, molecules, or ions) of a substance. The number 6.0×10^{23} is known as **Avogadro's number** (the number of atoms in exactly 12 g of C–12). Because 1 mole of any substance always contain 6.0×10^{23} particles, the mole is an ideal unit for chemical reactions. For example, 1 mole of carbon atoms (12 g of C) reacts with 1 mole of oxygen molecules (32 g of O_2) to produce 1 mole of carbon dioxide (44 g of CO_2). Remember that the mole ratio for chemical reactions is not always 1 to 1. For example, 2 moles of H_2 react with 1 mole of O_2 to give 2 moles of H_2O (here, the ratio is 2 : 1).

$$2H_2 + O_2 \rightarrow 2H_2O$$

Molar mass is the mass (in grams) of one mole of an element. As an example, let us express the relationship between the mole, the molecular mass, the molar mass, and Avogadro's number in the context of sucrose (sugar):

$$\text{Mole of sucrose} = \text{Molecular mass of sucrose (342.3 g)} = \text{Molar mass of sucrose (342.3 g)} = 6.02 \times 10^{23} \text{ Molecules of sucrose}$$

Because molecular mass of sucrose is 342.3 g, one mole of sucrose ($C_{12}H_{22}O_{11}$) equals 342.3 g, so the molar mass of sucrose is also 342.3 g. In the same way, the molecular mass of calcium (Ca) is 40 g, and its molecule consists of one atom of calcium (Ca); therefore, one mole of calcium is 40 g. One molecule of oxygen gas consists of two atoms of oxygen (O), so its mole and molar mass is equal to its molecular mass (16 g) times two, equal to 32 g.

The number (amount) of mole (n) of a substance in a compound is given by

$$\text{Mole} = \frac{\text{Mass}}{MM} \tag{12.3}$$

The **mole fraction** (X) is the ratio of the moles of one component of a mixture (e.g., component M) to the total number of moles of all components. It is abbreviated X with a subscript to identify the component. The equation for mole fraction is

$$X_{\text{M}} = \frac{\text{Mole}_{\text{M}}}{\text{Total moles of all components}} \tag{12.4}$$

EXAMPLE 3

How many moles are in 513 g of sucrose?

$$\frac{\text{Mass}_{\text{Sucrosea}}}{MM_{\text{Sucrose}}} = \frac{513}{342.3} = 1.5 \text{ Mole}$$

EXAMPLE 4

How many moles of oxygen gas (O_2) would be generated from decomposition of 86.5 ml of 10% hydrogen peroxide (H_2O_2)?

$$2H_2O_2 \rightarrow 2H_2O + O_2$$

$$(86.5 \text{ ml } H_2O_2)\left(\frac{10 \text{ g } H_2O_2}{100 \text{ ml } H_2O_2}\right)\left(\frac{1 \text{ Mole } H_2O_2}{34.0 \text{ g } H_2O_3}\right)\left(\frac{1 \text{ Mole } O_2}{2 \text{ Mole } H_2O_2}\right) = 0.13 \text{ Mole } O_2$$

EXAMPLE 5

Given:
A solution containing 66.74 g of sugar and 33.26 g of water

Calculate:
The mole fraction of sucrose (X_{Sucrose}) in the solution

The number of mole of sucrose ($MM_{\text{Sucrose}} = 342.3$) and water ($MM_{\text{Water}} = 18$ g) in the solution can be calculated by Eq. (12.3) as

$$\text{Mole} = \frac{\text{Mass}}{MM_{\text{Sucrose}}} = \frac{66.74}{342.3} = 0.19 \quad \text{Mole} = \frac{\text{Mass}}{MM_{\text{Water}}} = \frac{33.26}{18.0} = 1.85$$

The mole fraction of sucrose is can be calculated by Eq. (12.4)

$$X_{\text{Socrose}} = \frac{0.19}{0.19 + 1.85} = 0.09$$

EQUIVALENT MASS AND NUMBER OF EQUIVALENT

An **equivalent mass** (*Eq*, or equivalent weight, gram equivalent, on simply equivalent) describes the **molecular mass** (*MM* or molecular weight) of elements and compounds (in grams) with respect to their **number of charges** (N_C). An equivalent mass (*Eq*) is given as

$$Eq = \frac{MM}{N_C} \tag{12.5}$$

If the number of charges (also called **valance**) of an ion is ±1, its *Eq* equal to its molecular mass. For example, 1 *Eq* of sodium ion (Na^+) is 23 g. If N_C of an ion is ±2, its *Eq* is equal to its molecular mass (in grams) divided by 2. Therefore, one *Eq* of Ca^{2+} is 40/2 = 20 g.

Milliequivalent (*mEq*) is used when the concentration is too low. In sugar technology, *mEq* is used for expressing hardness (limesalts) content in molasses (see Chapters 6 and 8). *mEq* is given as

$$mEq = \frac{Eq}{1000} \tag{12.6}$$

Because different atoms or molecules have different masses, substances react not on the basis of their molecular masses but on their equivalent masses. If we were to measure 4 g of hydrogen and 32 g of oxygen, both would have the same number of atoms. So, we can say that they are weighed based on their **equivalent-mass ratio**. So, four molecules of hydrogen (*MM* of H is 1 g) combine with two molecules of oxygen (*MM* of O is 16 g) to form two molecules of water (*MM* = 18 g):

$$2H_2 + O_2 \rightarrow 2H_2O$$

This reaction tells us that two moles of H_2 (4 g) react with one mole of O_2 (32 g) to produce two moles of H_2O (36 g). Therefore, the equivalent mass relationship is 2 to 1. Remember that the difference in the number of charges (N_C) between hydrogen and oxygen is the reason that

we need two equivalent masses of hydrogen to react with one equivalent mass of oxygen (the N_C of hydrogen is 1 and that of oxygen is 2).

The **number of equivalents** (N_{Eq}) is given as

$$N_{Eq} = \frac{\text{Mass}}{MM/N_C} \tag{12.7}$$

For example, the number of equivalent of an acid should be equal to the number of equivalent of a base to neutralize each other (see Example 35 in this section). The following Notes and Examples the subject of equivalent mass and the number of equivalent.

NOTES

- **The number of charges (N_C) of acids is equal to the number of hydrogen ions (H^{+1}) they can furnish. For example, N_C of hydrochloric acid (HCl) is 1 because 1 mole of HCl can furnish 1 mole of hydrogen ion. The N_C of sulfuric acid (H_2SO_4) is 2 because it can furnish 2 moles of hydrogen ion.**
- **The number of charges (N_C) of bases is equal to the number of hydroxyl ions (OH^-) they can furnish. For example, N_C of sodium hydroxide (NaOH) is 1 because it can furnish 1 mole of hydroxyl ion. N_C of calcium hydroxide [$Ca(OH)_2$] is 2 because it can furnish 2 moles of hydroxyl.**

EXAMPLE 6

Ammonia (NH_3) releases 1 H^+ when it reacts with water according to the following reaction. Therefore, the equivalent mass in grams of ammonia is the same as its molecular mass (17 g), because $N_C = 1$ and its equivalent mass is

$$NH_3 + H_2O \rightarrow NH_4OH \quad Eq = \frac{17}{1} = 17\,\text{g equivalent}$$

Eq of **sulfuric acid** (H_2SO_4) is equal to its molecular mass (98 g) divided by 2 because 1 mole of this acid can furnish 2 moles of hydrogen ion (H^+), and its equivalent mass is

$$Eq = \frac{98}{2} = 49\,\text{g}$$

Calcium oxide (CaO) consists of calcium and oxygen. The number of charges (N_C) of both elements is 2 (Ca^{2+} is positively charged and O^{-2} has negative charges). $MM_{CaO} = 56$, so the equivalent mass of CaO is

$$Eq = \frac{MM}{N_C} = \frac{56\,\text{g}}{2} = 28\,\text{g}$$

Calcium hydroxide [$Ca(OH)_2$] contains 2 OH^- per molecule, which can be replaced. Its molecular mass is 74.0. Therefore, $N_C = 2$ and its equivalent mass is

$$Eq = \frac{74}{2} = 37\,\text{g}$$

Sodium carbonate (Na_2CO_3), or soda ash, contains two atoms of Na^+ per molecule, which can be replaced. Its molecular mass is 106 g. Therefore, $N_C = 2$, and its equivalent mass is

$$Eq = \frac{106}{2} = 53\,\text{g}$$

EDTA.2H$_2$O (ethylendiamine tetraacetic acid dihydrate) can make two bonds to a metal ion (e.g., calcium). Therefore, its N_C is 2, and its equivalent mass is

$$Eq = \frac{372}{2} = 186\,\text{g}$$

Aluminum hydroxide [Al(OH)$_3$] contains three hydroxyl ions per molecule. Therefore, its N_C is 3, and its equivalent mass is

$$Eq = \frac{78}{3} = 26\,\text{g}$$

EXAMPLE 7

Calculate the **number of equivalent** (N_{Eq}) in 20.0 g NaOH.

As previously seen, NaOH has $N_C = 1$ and a molecular mass (MM) of 40, so its number of equivalent (N_{Eq}) can be calculated by Eg. (12.7)

$$N_{Eq} = \frac{\text{Mass}}{MM/N_C} = \frac{20.0}{(40.0/1)} = 0.5$$

EXAMPLE 8

Given:
100 ml of juice containing 2% (m/v) Ca^{2+}

Calculate:
The number of equivalent (N_{Eq}) of Ca^{2+} in this juice in *mEq*.

MM of Ca is 40.0 g. 100 ml of this juice contains 2 g Ca^{2+}, so its N_{Eq} is

$$N_{Eq} = \frac{\text{Mass}}{MM/N_C} = \frac{2.0}{(40.0/2)} = 0.1\,Eq \quad 0.1 \times 1000 = 100\,mEq$$

CONCENTRATION OF SOLUTIONS

The **concentration** of a solution expresses the amount of solute (dissolved solid) in that solution. In other words, concentration means mass per unit volume. Concentrations in chemistry are expressed in several ways, as described next.

Percent by Mass (% m/m)

Percent by mass (%m/m) or percent mass fraction (simply **mass fraction**) is one way of expressing the concentration of solutions. A solution with 80% by mass (by weight) dry substance

(*DS*) contains 80 g (or any other mass units, e.g., kg or lb) of *DS* (assume all *DS* is in dissolved form) in 100 g of solution. Mathematically, we can express % by mass as

$$\frac{\text{Mass}_{\text{Solute}}}{\text{Mass}_{\text{Solution}}} \times 100 \tag{12.8}$$

EXAMPLE 9

Given:
A solution containing 26.0 g of sucrose in 100.0 g of water

Calculate:
The percent by mass of **solute** (sucrose) in this solution

$$\frac{26.0}{26.0 + 100.0} \times 100 = 20.6\%\ \text{m/m}$$

The mass percentage of water is 100 − 20.6 = 79.4%.

NOTES

- **Refractometric dry substance (*RDS*) and polarimetric sucrose (*PS*), two frequently used terms in the sugar industry, are defined as percent by mass. A 10% *RDS* sucrose solution contains 10 g of sucrose per 100 g of solution, meaning that 10 g of granulated-refined (GR) sugar is mixed with 90 g of water to form 100 g of this solution.**
- **If the percent is not expressed in a certain dimension, it indicates percent by mass (% by mass fraction). For example, beet containing 0.3% raffinose means there is 0.3 kg (or any other mass units) raffinose per 100 kg (or any other mass units) of beet.**
- **The terms on and per, in mathematical notation are equivalent to divide by. For example, if a factory uses 345 t/day of limestone and processes 7865 t of sugarbeet in the same period, the percent of limestone usage is (345/7865)100 = 4.4% on beet (OB).**

Percent by Volume (% v/v)

Percent by volume (% v/v) is another way of expressing concentration. For example, a 20% by volume of ammonium hydroxide (NH_4OH) solution contains 20 ml of NH_4OH per 100 ml of solution. Note that this is not the same as mixing 20 ml of NH_4OH with 80 ml of water. We can express % by volume as

$$\frac{\text{Volume}_{\text{Solute}}}{\text{Volume}_{\text{Solution}}} \times 100 \tag{12.9}$$

EXAMPLE 10

What is the % by volume of an ammonium hydroxide solution prepared by mixing 100.0 ml of the hydroxide with enough water to make a total volume of 500 ml of solution?

$$\frac{100.0}{500.0} \times 100 = 20.0\%\ \text{v/v}$$

NOTE

In recent chemical literature, percent by volume is sometimes called % volume fraction or simply volume fraction (v/v).

Percent by Mass/Volume (% m/v)

Percent by mass/volume (% m/v) is the next expression of concentration. For example, a 20% m/v calcium chloride ($CaCl_2$) solution contains 20 g of $CaCl_2$ per 100 ml of solution. In other words, 20 g of $CaCl_2$ is mixed with water to form 100 ml of $CaCl_2$ solution. A 40.0% m/v of sodium hydroxide (NaOH) contains 40.0 g of NaOH per 100 ml of solution, meaning that 40.0 g of this chemical is mixed with water to make 100 ml of diluted caustic solution.

Parts per Million by Mass (ppm m/m)

Parts per million by mass (ppm m/m) is usually used when the concentration is very low. This refers to the grams of a substance in million (10^6) g of sample. For example, if a trace of iron ions (Fe^{2+}) in solid NaCl is stated as 5 ppm, 1 million g of NaCl contains 5 *g* of Fe^{2+} or, equivalently, 1000 g (1 kg) NaCl contains 5 mg of Fe^{2+}.

$$\text{ppm} = \frac{\text{Mass}_{\text{Component}}}{\text{Mass}_{\text{Solution}}} \times 10^6 \tag{12.10}$$

Parts per Million by Mass Volume (ppm m/v)

Parts per million by mass volume (ppm m/v) is the grams of a substance in one million milliliters (10^6 ml). This can be expressed also as mg/L of a sample. For example, if a wastewater pond contains 500 ppm of Na^+, the pond water contains 500 g Na^+ in one million ml (1000 L), or 500 mg Na in one 1000 ml (1 L).

Parts per Billion by Mass (ppb m/m)

Parts per billion by mass is used for solutions that are even more dilute than those that can be expressed by ppm. The ppb refers to the grams of a substance in billion (10^9) g of the sample.

Parts per Billion by Mass Volume (ppb m/v)

Parts per billion by mass volume (ppb m/v) is the grams of a substance in 10^9 ml of a sample.

Because concentration in chemistry is expressed in different ways, it is helpful to know how to convert from one to another. The following four examples should help you understand the conversions.

EXAMPLE 11

Given:

A 26% by mass (% m/m) sugar solution

Calculate:

Concentration of the solution by mass/volume (% m/v)

The 26% (m/m) sugar solution contains 26 g of sugar for each 74 g of water and its density is 1.11 g/ml (from Table A.1). Volume (V) is determined by the density (d) equation:

$$d = \frac{\text{Mass}}{V} \quad V = \frac{100}{1.11} = 90.1\,\text{ml}$$

$$\frac{\text{Mass}_{\text{Solute}}}{V_{\text{Solution}}} \times 100 = \frac{26.0}{90.1} \times 100 = 28.9\%\ \text{m/v}$$

EXAMPLE 12

How many grams of 20.0% by mass (% m/m) aluminum chloride ($AlCl_3$) solution is needed to contain 50 g $AlCl_3$?

By proportion, letting x = the required grams of $AlCl_3$ solution,

$$\frac{20.0\,\text{g AlCl}_3}{100\,\text{g Solution}} = \frac{50\,\text{g AlCl}_3}{x\,\text{g Solution}} \quad x = \frac{50 \times 100}{20.0} = 250\,\text{g}$$

EXAMPLE 13

Given:

Nitric acid (HNO_3) assay	70% by mass
Nitric acid density (d)	1.4 g/ml

Calculate:

Mass of pure HNO_3 per cm^3 (ml) of concentrated acid

Because 70% of the total mass of acid is pure HNO_3, the mass of pure HNO_3 in 1 ml of acid is

$$\text{Mass} = V \times d = 70 \times 1.4 = 98.0\,\text{g}/100\,\text{ml}\ \text{(or 0.1 g/ml)}$$

EXAMPLE 14

Aluminum chloride hexahydrate ($AlCl_3.6H_2O$) is used as a reagent to determine % sugar in cossettes and diffusion juice. How many grams of $AlCl_3.6H_2O$ are needed to prepare 1000 g of 20% $AlCl_3$?

A 20% $AlCl_3$ solution contains 20.0 g $AlCl_3$ per 100 g of solution. Therefore, we need 200 g of $AlCl_3$ to make 1000 g of solution. The molecular mass (*MM*) of $AlCl_3$ is 133.5, and that of $AlCl_3.6H_2O$ is 241.5. The amount of aluminum chloride can be calculated by proportion because 133.5 g $AlCl_3$ is in 241.5 g $AlCl_3.6H_2O$. Letting x = the required grams of $AlCl_3.6H_2O$,

$$\frac{133.5\,\text{g AlCl}_3}{241.5\,\text{g AlCl}_3.6\text{H}_2\text{O}} = \frac{1\,\text{g AlCl}_3}{x} \qquad x = \frac{1 \times 241.0}{133.0} = 1.8\,\text{g AlCl}_3.6\text{H}_2\text{O}$$

Because we need 200 g of $AlCl_3$ to prepare 1000 g of 20% $AlCl_3$,

$$200 \times 1.8 = 360\,\text{g AlCl}_3.6\text{H}_2\text{O}$$

NOTES

- **Thus far, you learned how to express concentration in percentages in different ways. In analytical chemistry, other important ways of expressing concentration exist. Three such concentrations are the following:**
 - **Molality (*m*)**
 - **Molarity (*M*)**
 - **Normality (*N*)**
- **Expressing the concentration of a solution by molality (*m*) is useful for calculating physical measurements (e.g., the boiling point or freezing point of sucrose solutions; see Section 4 of Chapter 1).**
- **Expressing the concentration by molarity (*m*) and normality (*N*) is useful in laboratory practices when preparing standard solutions (solutions with known and exact concentrations) and in the titration process (titration of a solution with unknown concentration with the help of a standard solution).**

MOLALITY

Molality (*m*, also known as **molal concentration**) is the concentration of a solution expressed as the number of moles of solute (n) in 1 kg of solution. The molality of a solution is given as

$$m = \frac{n}{\text{kg}_{\text{Solution}}} = \frac{\text{Mass}_{\text{Solute}}}{MM_{\text{Solute}} \times \text{kg}_{\text{Solution}}} \tag{12.11}$$

MM_{Solute} Molecular mass (weight) of solute

The mass of a substance needed to prepare a solution with a given molality is calculated using the following equation, which is derived from Eq. (12.11):

$$\text{Mass}_{\text{Solute}} = m \times MM_{\text{Solute}} \times \text{kg}_{\text{Solution}} \tag{12.12}$$

For example, a solution containing 58.5 g of NaCl per 1 kg of solution is a 1 molal (1 *m*) solution. Or a solution made up of one mole of sulfuric acid (one mole of sulfuric acid weighs 98.0 g) dissolved in water to prepare 1 kg solution is a 1.0 molal solution of H_2SO_4.

EXAMPLE 15

Given:

A solution contains 35.5 g of sugar (the sucrose has a molecular mass of 342.3) dissolved in 155 g (0.155 kg) of water

Calculate:

The molality (*m*) of this solution

$$m = \frac{\text{Mass}_{\text{Solute}}}{MM_{\text{Solute}} \times \text{kg}_{\text{Solution}}} = \frac{35.5}{342.3 \times 0.155} = 0.67 \text{ mole/kg} \text{ (or } 0.67\,m\text{)}$$

NOTES

- **In a molal solution, the total mass of the solution is known, while its volume is not known. Therefore, the molality cannot be calculated from the molarity unless the density of the solution is known.**
- **Molality (*m*) is used to calculate boiling-point elevation (*BPE*) and freezing-point depression (*FPD*). Refer to Section 4 of Chapter 1 for calculations.**

MOLARITY

Molarity (*M*, also known as **molar concentration**) is the concentration of a solution expressed as the number of moles of a substance (solute) in one liter (L) of solution. In other words, a solution containing 1 mole of solute in a total of 1 liter (L) solution is 1 molar (1 *M*). The molarity of a solution is given as

$$M = \frac{\text{Mole}_{\text{Solute}}}{\text{L}_{\text{Solution}}} = \frac{\text{Mass}_{\text{Solute}}}{MM_{\text{Solute}} \times \text{L}_{\text{Solution}}} \tag{12.13}$$

L Number of liters of solution

The mass of a substance needed to prepare a solution of a given volume and molarity can be calculated using the following equation derived from Eq. (12.13):

$$\text{Mass}_{\text{Solute}} = M \times MM_{\text{Solute}} \times L_{\text{Solution}} \tag{12.14}$$

For example, a solution containing 58.5 g of NaCl ($MM_{\text{NaOH}} = 58.5$) dissolved in water to prepare 1 L is 1 molar (1 *M*) solution. Or, a 2.0 *M* solution of NaOH ($MM_{\text{NaOH}} = 40$ g) contains 2 moles, or $2 \times 40 = 80$ g of NaOH per liter of solution.

NOTE

- **A solution of a given molarity is prepared by dissolving the solute in enough water to give a final volume of 1 liter (not by dissolving it in an initial volume of 1 L).**

EXAMPLE 16

Determine the molarity of 20.0 g $AlCl_3.6H_2O$ (its $MM = 241.5$) per 100 ml of solution. This solution is used in the sugar laboratory as a clarifying reagent.

$$M = \frac{\text{Mass}_{\text{Solute}}}{MM_{\text{Solute}} \times \text{L}_{\text{Solution}}} = \frac{20.0}{241.5(100/1000)} = 0.8\,\text{mole/L (or } 0.8\,M)$$

EXAMPLE 17

Determine the molarity of hydrogen ions (H^+) in a solution of HCl in 250 ml of water to produce 0.10 g of H^+ ($MM_H = 1.0$) in the resulting solution.

Because the moles of H^+ equal its grams, the number of moles of H^+ in the sample is 0.10:

$$M = \frac{\text{Mass}_{\text{Solute}}}{MM_{\text{Solute}} \times \text{L}_{\text{Solution}}} = \frac{0.10}{1.0(250/1000)} = 0.4\,\text{mole/L (or } 0.4\,M)$$

EXAMPLE 18

Calculate the molarity (M) of dissolved oxygen per liter of dilution water for a BOD (biochemical oxygen demand) test if 1 L of water contains 8.2 mg (0.0082 g) of dissolved oxygen.

As noted previously, oxygen belongs to a class of diatomic substances, which have two atoms in their molecules (O_2). Therefore, its mole is equal to double of its molecular mass ($2 \times 16 = 32$ g):

$$M = \frac{\text{Mass}_{\text{Solute}}}{MM_{\text{Solute}} \times \text{L}_{\text{Solution}}} = \frac{0.0082}{32 \times 1} = 2.6 \times 10^{-4}\ \text{mole/L (or } 0.00026\,M)$$

EXAMPLE 19

EDTA.2H_2O (ethylendiamine tetraacetic acid dihydrate) is used to determine **hardness** (limesalts) in different in-process products. How many grams of EDTA.2H_2O, with a molecular mass (MM) of 372, are needed to prepare 1 L of a 0.018 M solution?

$$\text{Mass}_{\text{Solute}} = M \times MM_{\text{Solute}} \times \text{L}_{\text{Solution}} = 0.018 \times 372 \times 1 = 6.7\,\text{g}$$

EXAMPLE 20

How many grams of sucrose, $C_{12}H_{22}O_{11}$ ($MM_{Sucrose}$ = 342.3 g/mole) should be dissolved in water to make 1 L of 0.5 *M* sucrose solution?

$$\text{Mass}_{\text{Solute}} = M \times MM_{\text{Solute}} \times \text{L}_{\text{Solution}} = 0.5 \times 342.3 \times 1 = 171\,\text{g}$$

EXAMPLE 21

Calculate the molarity of sucrose normal solution (26.00 g of sucrose dissolved in water to a final volume of 100 ml), $MM_{Sucrose}$ = 342.3 g.

You need 260.00 g of sucrose dissolved in water to a final volume of 1 L to make 1 L of sucrose normal solution, so the molarity of sucrose is

$$M = \frac{\text{Mass}_{\text{Solute}}}{MM_{\text{Solute}} \times \text{L}_{\text{Solution}}} = \frac{260.00}{342.3 \times 1} = 0.76\ \text{mole/L}\ \ (\text{or } 0.76\,M)$$

EXAMPLE 22

Given:

Concentration of a glucose ($C_6H_{12}O_6$) solution 0.10 *M*
$MM_{glucose}$ = 180 g/mole

Calculate:

The number of moles and liters of 0.10 *M* glucose solution to provide 110 g of glucose

The number of moles in 110 g is calculated by Eq. (12.3)

$$\text{Mole} = \frac{\text{Mass}_{\text{Glucose}}}{MM_{\text{Glucose}}} = \frac{110}{180} = 0.6$$

$$M = \frac{\text{Mass}_{\text{Solute}}}{MM_{\text{Solute}} \times \text{L}_{\text{Solution}}} \qquad \text{L}_{\text{Solution}} = \frac{\text{Mass}_{\text{Solute}}}{MM_{\text{Solute}} \times M} = \frac{110}{180 \times 0.1} = 6.1$$

NORMALITY

Normality (*N*) is the concentration of a solution expressed as the number of equivalent mass (*Eq*) of substance (solute) in 1 liter of solution. The normality of a solution is calculated using the equivalent mass of solute (Eq_{Solute}):

$$N = \frac{\text{Mass}_{\text{Solute}}}{Eq_{\text{Solute}} \times \text{L}_{\text{Solution}}} \tag{12.15}$$

The mass of substance needed to prepare a solution of a given volume and normality is calculated using the following equation, which is derived from Eq. (12.15):

$$\text{Mass}_{\text{Solute}} = N \times Eq_{\text{Solute}} \times \text{L}_{\text{Solution}} \tag{12.16}$$

NOTE

- **The normality may vary appreciably as the temperature is changed because volume is temperature dependent.**

EXAMPLE 23

Calculate the normality of HCl in its concentrated form. Hydrochloric acid (muriatic acid) usually has an assay (concentration) of 36% (by mass), which has a density of 1.18. The equivalent mass of the HCl is the same as its molecular mass (36.5 g).

By using the density (d) equation, we can calculate the volume (V) of 100 g of the HCl solution:

$$d = \frac{\text{Mass}}{V} \quad V = \frac{\text{Mass}}{d} = \frac{100}{1.18} = 84.7\,\text{ml or } 0.0847\,\text{L}$$

By the normality equation,

$$N = \frac{\text{Mass}_{\text{Solute}}}{Eq_{\text{Solute}} \times \text{L}_{\text{Solution}}} = \frac{36}{36.5 \times 0.0847} = 11.6\ \text{Normal (or } 11.6\,N)$$

EXAMPLE 24

Calculate how many grams of EDTA.2H_2O (molecular mass, MM = 372) are needed to prepare 1 L of a 0.036 N of this solution, which is used in hardness (limesalts) determination in the sugar lab.

The number of charges (N_C) of EDTA is equal to 2. By the equivalent mass (Eq) equation [(Eq. 12.5)]:

$$Eq = \frac{MM}{N_C} = \frac{372}{2} = 186$$

The mass of EDTA needed to prepare a 0.036 N solution is

$$\text{Mass}_{\text{Solute}} = N \times Eq_{\text{Solute}} \times \text{L}_{\text{Solution}} = 0.036 \times 186 \times 1 = 6.7\,\text{g}$$

So we must dissolve 6.7 g of EDTA.2H_2O in DI (deionized) water and adjust the volume to exactly 1 L to have 0.036 N EDTA.2H_2O.

EXAMPLE 25

Given:

Ammonium hydroxide (NH_4OH) is used as a reagent to determine % sugar in cossettes and the diffusion juice of the sugar factory. The equivalent mass (Eq) of ammonium hydroxide is 35. It usually comes as an aqueous solution with 28% m/m assay (as NH_3) and a density of 0.9 g/ml.

Calculate:

Normality (N) of the reagent in its concentrated form
Normality of 20% of this reagent

By using the density equation, we can calculate the volume of 100 g of the NH_4OH solution by density equation:

$$d = \frac{\text{Mass}}{V} \quad V = \frac{M}{d} = \frac{100}{0.989} = 101.1\,\text{ml, or } 0.10\,\text{L}$$

By the normality equation:

$$N = \frac{\text{Mass}_{\text{Solute}}}{Eq_{\text{Solute}} \times \text{L}_{\text{Solution}}} = \frac{28}{35 \times 0.10} = 8.0\ \text{Normal}$$

The 20% ammonium hydroxide is made by diluting 20 ml of the concentrated reagent to a total volume of 100 ml. Therefore, the normality of the reagent is reduced by 5,

$$N = \frac{8.0}{5} = 1.6\ \text{Normal}$$

Normality (N) can be related to **molarity** (M) by using the following equation:

$$N = N_C \times M \tag{12.17}$$

EXAMPLE 26

What is the normality of a 0.036 M solution of EDTA? The number of charges (N_C) of EDTA is 2.

$$N = N_C \times M = 2 \times 0.036 = 0.072$$

EXAMPLE 27

Given:

Concentration (assay) of phosphoric acid (H_3PO_4)	85.0% (m/m)
Density of H_3PO_4 solution	1.71 g/ml
Molecular mass of H_3PO_4	98.0 g/mole

Calculate:
The normality (N) of this acid

By using the density equation, you can calculate the volume (V) of 100 g of the H_3PO_4 solution:

$$V = \frac{\text{Mass}}{d} = \frac{100}{1.71} = 58.48\,\text{ml, or } 0.05848\,\text{L}$$

By the molarity (M)equation,

$$M = \frac{\text{Mass}}{MM \times \text{L}_{\text{Solution}}} = \frac{85}{98.0 \times 0.05848} = 14.8\,\text{Molar (or } 14.8\,M)$$

H_3PO_4 contains three acid equivalents per mole; therefore, $N_C = 3$. By the normality-molarity equation,

$$N = N_C \times M = 3 \times 14.8 = 44.4\ \text{Normal (or } 44.4\,N)$$

CHEMICAL FORMULA AND CHEMICAL EQUATION

A **chemical formula** (molecular formula) gives the symbol and number of the atoms of each compound. For example, a molecule of **sucrose** (sugar) consists of 12 atoms of carbon, 22 atoms of hydrogen, and 11 atoms of oxygen, and its molecular formula is $C_{12}H_{22}O_{11}$.

A **chemical equation** is a shorthand way of expressing a chemical reaction by using its chemical formulas. The following equation describes the decomposition of calcium carbonate ($CaCO_3$) in the limekiln of a beet-sugar factory to produce lime (CaO) and carbon dioxide gas (CO_2):

$$CaCO_3 \rightarrow CaO + CO_2$$

In this equation, $CaCO_3$ is the **reactant** (it appears to the left of the arrow in a chemical equation) and CaO and CO_2 are the **products**. The arrow is a symbol of change. In this reaction, substances are changed (converted) into other substances that have different compositions and structures. As a result of this reaction, the following changes took place:

- Bonds were broken
- Atoms were reorganized
- New compounds were formed

Stoichiometry (pronounced **sto-keo-metry**) is about the quantitative relations between the reactants and products in a chemical reaction. When chemicals react, they are mixed in balanced **stoichiometric quantities**. A balanced equation has the same number and type of atoms on both sides of the equation. Consider a typical reaction used in sugar labs in the alkalinity test. In this test, the alkalinity of first- and second-carb juice is determined by titrating the juice sample with sulfuric acid according to the following balanced equation:

$$H_2SO_4 + CaO \rightarrow CaSO_4 + H_2O$$
$$98\,\text{g} + 56\,\text{g} \rightarrow 136\,\text{g} + 18\,\text{g}$$
$$154\,\text{g} \rightarrow 154\,\text{g}$$

Remember, the equations are balanced by introducing coefficients, and at the end, the number of atoms of each compound should be the same on both sides of the equation. For example,

$$N_2 + 3H_2 \rightarrow 2NH_3$$

NOTES

- **In compounds, the number of cations (positively charged ions) and anions (negatively charged ions) are the same. In a chemical formula, the sum of the positive and negative numbers must equal 0. For example, in aluminum chloride ($ALCl_3$), three chloride ions (Cl^-), each with one negative charge, balance one aluminum ion (Al^{3+}) with three positive charges.**
- **In chemical formulas, the cation (+) ion is written before the anion (–) ion. For example, in H_2SO_4 the cation (H^+) is written in front of anion (SO_4^{2-}). There are two exceptions, in which the anion is written before the cation. These are ammonia (NH_3) with (N^{3-}) and (H^+), and methane (CH_4) with C^{4-} and H^+.**
- **Following are common names of several chemicals used in sugar technology:**
 - **Borax = Sodium borate ($Na_2B_4O_7$)**
 - **Caustic = Sodium hydroxide (NaOH)**
 - **Potash = Potassium carbonate (K_2CO_3)**
 - **Caustic potash = Potassium hydroxide (KOH)**
 - **Soda ash = Sodium carbonate (Na_2CO_3)**
 - **Muriatic acid = Hydrochloric acid (HCl)**
 - **Limestone = Calcium carbonate ($CaCO_3$)**
 - **Milk of lime = Calcium hydroxide [$Ca(OH)_2$]**
 - **Baking soda = Sodium bicarbonate ($NaHCO_3$)**
 - **Quicklime (simply lime) = Calcium oxide (CaO)**
 - **Gypsum = Calcium sulfate dihydrate ($CaSO_4.2H_2O$)**
- **Chemists write the formula of hydrates with a centered dot to separate the two parts of the formula. For example, the formula of gypsum is written as $CaSO_4.2H_2O$ (not $CaSO_4.2H_2O$).**

ACID AND BASE

An **acid** is a substance that produces hydrogen ions (H^+) when it is dissolved in water, such as **hydrochloric acid** (HCl). A **base** is a substance that produces hydroxide ions (OH^-) when dissolved in water, such as, **sodium hydroxide** (NaOH).

Vinegar, citrus fruit, aspirin, and vitamin C are acidic. Milk of magnesia and baking soda are basic. Water has both acidic and basic properties because its molecules do not dissociate into H^+ and OH^-.

Acids and bases can be classified as strong or weak depending on the ability of their molecules to ionize (dissociate or decompose) in water. For example, nitric acid (HNO_3) is completely ionized in water into hydrogen ions (H^+) and nitrate ions (NO_3^-), so it is in the strong-acid group. So are hydrochloric (HCl) and sulfuric (H_2SO_4) acids. Acetic acid (vinegar acid, HCH_3COO) and boric acid (H_3BO_3) are in the weak-acid group because they are only partly ionized. When acetic acid reacts with water, only 1% of its molecules are ionized into H^+.

In this respect, even a low concentration of a strong acid (such as nitric or hydrochloric acid) damages the skin, but a weak acid such as **boric acid** (H_3BO_3) can be used as a medicine even in eyes.

Sodium hydroxide (NaOH) is completely ionized in water to hydroxyl ions (OH^-) and sodium ions (Na^+), so it is a strong base. Potassium hydroxide (KOH) is also a strong base. On the other hand, calcium hydroxide [$Ca(OH)_2$] is a weak base because it is only partially ionized.

The **pH scale** was invented to determine the acidity or basicity of solutions. The pH scale is calibrated so that pure water has a pH value of 7. Acids have a pH from 1 to 7 (*the higher the pH, the weaker is the acid*). Bases (alkalis) have a pH from 7 to 14 (*the higher the pH, the stronger the base*) (for more information, refer to the pH meter subsection in Section 2 of Chapter 11).

NOTES

- **Salt (not table salt) is a general term meaning any ionic compound produced from the reaction between an acid and a base. Note that salts are far less corrosive than the acids and bases from which they are produced.**
- **Ammonia (NH_3) is a common base, although it does not contain an OH^- ion. When ammonia is added to water, it takes a H^+ ion from the water molecule and produces an OH^- ion:**

$$NH_3 + H_2O \rightarrow NH_4^+ + OH^-$$

This is the main reason for the pH drop of the juice during evaporation in the sugar factory because ammonia is vented out from the evaporator bodies.

STANDARD SOLUTION PREPARATION

A standard solution (a solution with a known and exact concentration) can be prepared by diluting a **stock reagent** (a purchased or prepared concentrated solutions) with water. In general, the amount of **solute** (dissolved solid) contained in a given volume of a solution is equal to

$$\text{Amount of solute} = \text{Volume} \times \text{Concentration}$$

or

$$\text{Amount of solute} = V \times C \tag{12.18}$$

When the stock solution is diluted with water, the volume (V) is increased and the concentration (C) is decreased, but the amount of solute remains the same because water contains no solid. Since the mass of the solute in the solutions remains unchanged, before dilution (indicated as 1) and after dilution (2), we can establish a general equation for dilutions:

$$V_1 \times C_1 = V_2 \times C_2 \tag{12.19}$$

Since **normality** (N) is one of the ways of expressing concentration, it can be substituted for concentration in the preceding equation:

$$V_1 \times N_1 = V_2 \times N_2 \tag{12.20}$$

Any of items in Eqs. (12.19) and (12.20) can be calculated if the other three are known.
Usually, chemicals come from suppliers in labeled bottles indicating the molecular mass (MM), density (d), and the percentage assay (concentration, C). To prepare a solution of the desired concentration (desired normality [N] or molarity [M]) from these stocks, the following equations are used:

EXAMPLE 28

Given:
A 5.0 normal solution of sodium hydroxide (NaOH)

Calculate:
How many ml of this stock are required to make 1 L (1000 ml) of 0.2 *N*

Let 5.0 *N* hydroxide be solution 1 and 0.2 *N* be solution 2. Then,

$$V_1 \times N_1 = V_2 \times N_2 \quad V_1 = \frac{V_2 \times N_2}{N_1} = \frac{1000 \times 0.2}{5.0} = 40\,\text{ml}$$

Therefore, we need 40 ml of 5.0 *N* NaOH to be diluted with DI water to make 1 L of 0.2 *N* NaOH.

$$V = \frac{100 \times MM \times N}{C \times d \times N_C} \tag{12.21}$$

Because $N = N_C \times M$,

$$V = \frac{100 \times MM \times M}{C \times d} \tag{12.22}$$

N_C Number of charges (valence)

EXAMPLE 29

Given:

Concentration (*C*) of H_2SO_4	96.0% (by mass)
Density (*d*) of H_2SO_4	1.84 g/ml
Molecular mass (*MM*) of H_2SO_4	98.0 g/mole

Calculate:
How many ml of H_2SO_4 is needed to prepare 1 L of 0.36 *N* of sulfuric acid solution
How many ml of H_2SO_4 is needed to prepare 1 L of 0.07 *N* of sulfuric acid solution
The percentage of 0.07 *N* H_2SO_4

$$V = \frac{100 \times MM \times N}{C \times d \times N_C} = \frac{100 \times 98.0 \times 0.36}{96.0 \times 1.84 \times 2} = 10.0\,\text{ml}$$

Therefore, we have to dilute 10 ml of concentrated sulfuric acid with DI water into a 1-L flask and fill it to the mark of the flask to make 1 L of 0.36 *N* H_2SO_4 solution.

To produce 1 L of 0.07 *N* sulfuric acid, we need

$$V = \frac{100 \times MM \times N}{C \times d \times n} = \frac{100 \times 98.0 \times 0.07}{96.0 \times 1.84 \times 2} = 1.9\,\text{ml}$$

The 1.9 ml is in 1 L of the acid solution, so the percentage of the solution is

$$1.9 \div 10 = 0.19\%$$

EXAMPLE 30

Prepare 1000 ml of 1.0 *N* hydrochloric acid reagent from concentrated hydrochloric acid. The HCl usually comes in an assay (concentration) of 37% m/m. The molecular mass of HCl is 36.5, and its 37% solution has a density of 1.2 ml/g.

$$V = \frac{100 \times MM \times N}{C \times d \times N_C} = \frac{100 \times 36.5 \times 1.0}{37 \times 1.2 \times 1} = 82.2\,\text{ml}$$

Therefore, we have to dilute 82.2 ml of stock solution with DI water into a 1-L flask and fill it to the mark of the flask to make 1 L of 1.0 *N* HCL solution.

TITRATION

Titration is based on the addition of a **standard solution** (**titrant**, a solution with known concentration) to a solution of unknown concentration, until a certain pH value is obtained. The point of neutralization is called the **endpoint**. Then, the unknown concentration can be calculated from the titrant concentration and its used volume.

To perform this type of measurement, you need an **indicator**, which is a substance that changes the color of the solution at a certain pH. Ideally, *an indicator should indicate a sharp change in color over a narrow pH range*. Examples are **phenolphthalein** (with a pH range of 8 to 10) and **methyl red** (with a pH range of 4.4 to 6.2).

In a titration test, a standard solution (titrant) is delivered by a burette to a dish containing a known amount of a solution with an unknown concentration. An indicator is used to find the **endpoint of the titration** (the point where two reactants are chemically equivalent and complete).

The number of equivalents of the known base or acid added is calculated by multiplying the volume by its normality (*N*). Because one equivalent of an acid is required to neutralize one equivalent of a base, the number of equivalents of the known should be equal to the number of equivalents of the unknown.

The followings are two examples of titration:

$$H_2SO_4 + 2NaOH \rightarrow Na_2SO_4 + H_2O$$

$$HCl + NaHCO_3 \rightarrow NaCl + CO_2 + H_2O$$

For titration involving acids with one hydrogen ion (H^+) per formula and bases with one hydroxide ion (OH^-), the number of moles of acid is identical to the number of moles of base required to neutralize the acid:

$$\text{Moles acid} = \text{Moles base} \times \frac{1\,\text{Mole acid}}{1\,\text{Mole base}} \tag{12.23}$$

EXAMPLE 31

Given:
In the titration of 10.0 ml of an unknown concentration of a hydrochloric acid sample, 19.6 ml of 0.3 *M* sodium hydroxide was used.

Calculate:
Number of mole of base and acid by Eq. (12.13)

$$M = \frac{\text{Mole}_{\text{Solute}}}{\text{L}_{\text{Solution}}} \quad \text{Mole}_{\text{NaOH}} = M_{\text{NaOH}} \times \text{L}_{\text{Solution}} = 0.3 \times \frac{19.6}{1000} = 5.9 \times 10^{-3}$$

$$\text{Moles acid} = \text{Moles base} \times \frac{1\ \text{Mole acid}}{1\ \text{Mole base}} = 5.9 \times 10^{-3} \times \frac{1}{1} = 5.9 \times 10^{-3}$$

EXAMPLE 32

Given:
A titration required 8.5 ml of 1.78 *M* NaOH solution.

Calculate:
How many moles of NaOH were in this volume?

By Eq. (12.13)

$$M = \frac{\text{Mole}_{\text{Solute}}}{\text{L}_{\text{Solution}}} \quad \text{Mole}_{\text{NaOH}} = M_{\text{NaOH}} \times \text{L}_{\text{Solution}} = 1.78 \times \frac{8.5}{1000} = 0.015$$

To perform a titration, proceed as follows:

1. Fill the buret with the titrant (standard solution).
2. Measure a certain amount of the sample (e.g., 20 ml) with a pipette and transfer it into a titration flask.
3. Add 2 to 3 drops of an appropriate **indicator** (see the Note) to the flask.
4. Add the titrant to the titration flask slowly while swirling the flask with one hand until the **endpoint** (the point at which the indicator changes color) is obtained. (Reduce the volume of the additions as the titration progresses. At the very end, the titrant should be added a drop at a time.)

NOTES

- **Indicators are usually organic dye, and their color is pH sensitive. A few drops (in the case of a liquid indicator) or few shakes (in the case of a powder indicator) are usually needed to do the job. For example, the indicator phenolphthalein is colorless in acidic solutions and pink in basic solutions. Methyl red is red in acid and yellow in base.**
- **The reagents used in sugar labs are prepared so that their concentrations are known. If an adjustment is needed after the titration for standardization, the additional amount of the acid or water must be calculated. The following examples are helpful.**

EXAMPLE 33

0.36 *N* sulfuric acid reagent is used in the sugar lab for determination of CaO in different juices. 1 L of this acid was prepared by diluting 9.8 ml of stock (concentrated) acid with DI water to 1 L. For standardization, 10.0 ml of prepared reagent was titrated with 0.36 *N* Na_2CO_3 standard solution. After the titration, an adjustment was needed to come up with the exact normality of 0.36 *N* solution.

Calculate:

The adjusted reagent if the amount of standard solution used for titration was 10.4 ml.
The adjusted acid solution if the amount of standard solution used was 9.7 ml.

In the first situation, the reagent is stronger than the desired one because the amount of used standard solution ($V_1 = 10.4$ ml) was larger than the amount of the sample ($V_2 = 10.0$ ml). Therefore, we need to add a certain amount of water to the solution to correct its normality to 0.36 (N_1). Let $V_1 = 10.4$, $N_1 = 0.36$ be the volume and normality of the standard solution, respectively. $V_2 = 10.0$, and N_2 will be the volume and normality, respectively, of the prepared reagent. Then,

$$V_1 \times N_1 = V_2 \times N_2 \quad 10.4 \times 0.36 = 10.0 \times N_2 \quad N_2 = 0.37$$

We can use the same equation to calculate the amount of water that has to be added to the reagent to reduce its normality to exactly 0.36. Let $V_2 = 990$ be the amount of prepared reagent left, because the first time the volume of the prepared reagent was 1 L and 10.0 ml of this solution was used earlier for titration. The normality of the prepared reagent was found to be 0.37 (N_2). Let the total volume of the solution that we have to adjust be V_1 to make a solution with a normality of 0.36 (N_1).

$$V_1 \times N_1 = V_2 \times N_2 \quad V_1 = \frac{V_2 \times N_2}{N_1} = \frac{990 \times 0.37}{0.36} = 1018\,\text{ml}$$

Therefore, the amount of water we need to add to the solution is the difference:

$$1018 - 990 = 28\,\text{ml}$$

In the second case, the prepared reagent is weaker than the desired one because the amount of standard solution ($V_2 = 9.7$ ml) was less than the amount of the sample ($V_2 = 10.0$ ml). This time, we have to add a certain amount of acid to the solution to correct its normality to 0.36 (N_1). Again, we first find out the normality of the prepared reagent (N_2) from Eq. (12.20):

$$V_1 \times N_1 = V_2 \times N_2 \quad 9.7 \times 0.36 = 10.0 \times N_2 \quad N_2 = 0.35$$

The amount of stock (concentrated) acid used the first time to make the reagent was 9.8 ml (V_2) and the intention was to make a 0.36 *N* (N_2), but we prepared a solution with 0.35 normality (N_1). Let the volume of the acid that we have to adjust to be V_1:

$$V_1 \times N_1 = V_2 \times N_2 \quad V_1 = \frac{V_2 \times N_2}{N_1} = \frac{9.8 \times 0.36}{0.35} = 10.1\,\text{ml}$$

Therefore, the amount of acid we need to add to the solution is equal to the difference of 10.1 and the original amount of acid that we had in the solution (9.8 ml). (Note that we did not consider the amount of reagent used for titration because this is very small.)

$$10.1 - 9.8 = 0.3\,\text{ml}$$

Therefore, we have to add 0.3 ml more concentrated stock acid to the solution to bring its normality up to 0.36.

EXAMPLE 34

Nitric acid 1.78 *N* reagent (1.78 Steffen acid) is used in sugar-labs for determination of CaO in limecake (carbonation-lime residue). 10.0 ml of sodium hydroxide (NaOH) was titrated with 1.78 *N* Steffen nitric acid. The amount of acid used was 9.0 ml.

Calculate:

The normality of the NaOH

The adjustment to the hydroxide solution to come up with exactly a 1.78 *N* NaOH solution if we prepare 1 L of this base,

$$H_3NO_3 + 3NaOH \rightarrow Na_3NO_3 + 3H_2O$$

Let the 1.78 *N* acid be solution 1, and the NaOH solution 2. Again, by Eq. (12.20),

$$V_1 \times N_1 = V_2 \times N_2 \quad N_2 = \frac{V_1 \times N_1}{V_2} = \frac{10.0 \times 1.78}{9.0} = 1.98\,N$$

In the second case, we have to add water to the NaOH solution to bring its normality down to 1.78. The amount of NaOH left is equal to 990 ml because 10 ml of this solution were used earlier for titration with acid. Therefore, the total volume of solution is now 990 ml (V_1), and its normality is found to be 1.98 (N_1). We let the total volume of the solution that we have to adjust be V_2 to make a solution with normality of 1.78 (N_2). Again, we use Eq. (12.20),

$$V_1 \times N_1 = V_2 \times N_2 \quad V_2 = \frac{V_1 \times N_1}{N_2} = \frac{990 \times 1.98}{1.78} = 1101\,\text{ml}$$

The amount of water needed to add to the solution is

$$1101 - 990 = 111\,\text{ml}$$

EXAMPLE 35

Alkalinity of the first- and second-carb juice is determined by titration of 10.0 ml of filtered juice with 0.036 *N* sulfuric acid. In a test, 9.2 ml of acid was used to reach the endpoint of phenolphthalein.

Calculate:

The alkalinity of the juice (in g per 100 ml)

$$H_2SO_4 + CaCO_3 \rightarrow CaSO_4 + CO_2 + H_2O$$

$$CaCO_3 \rightarrow CaO + CO_2$$

As we said previously, a 0.036 *N* sulfuric acid solution contains 0.036 gram equivalent per liter. This means that 1000 ml of this solution contains 0.036 g equivalent, and 1 ml of it contains 0.000036 gram equivalent.

Gram equivalent (*Eq* or equivalent mass) of CaO is

$$Eq = \frac{MM}{N_C} = \frac{56}{2} = 28\,\text{g equivalent}$$

MM	Molecular mass
N_C	Number of charges

By Eq. (12.16),

$$\text{Mass}_{\text{Solute}} = N \times Eq_{\text{Solute}} \times \text{L}_{\text{Solution}} = 0.036 \times 28 \times 1 = 1\,\text{g/L (or 1 mg/ml)}$$

Therefore, 1 ml of 0.036 *N* sulfuric acid is equal to 1 mg of CaO in the juice.

Use Eq. (12.20) with an acid solution as solution 1 and a juice (containing lime) as solution 2:

$$V_1 \times N_1 = V_2 \times N_2 \quad N_2 = \frac{V_1 \times N_1}{V_2} = \frac{10.0 \times 0.036}{9.2} = 0.033\,\text{Normal}$$

By Eq. (12.16),

$$\text{Mass}_{\text{Solute}} = 0.033 \times 28 \times \frac{10.0}{100} = 0.092\,\text{Alkalinity (g/100ml)}$$

We can also express the alkalinity based on the relation of the acid with the amount of CaO in the juice because earlier we proved that 1 ml of 0.036 *N* sulfuric acid is equal to 1 mg (or 0.001 g) of CaO in the juice. Also because we used 9.2 ml of the acid for 10 ml of the sample.

$$\text{Alkalinity} = \text{ml}_{\text{Acid}} \times 0.001 \times \frac{100}{\text{ml}_{\text{Sample}}}$$

$$\text{Alkalinity} = 9.2 \times 0.001 \times \frac{100}{10} = 0.092\,\text{g/100 ml}$$

MENISCUS

Meniscus, in chemistry, is the curving of liquids at their surface in a narrow container. Because **water** and **glass** are polar substances, **adhesive force** (attractive force between molecules of two polar substances) between their molecules occurs if they are in contact with each other. As a result, some water molecules adjacent to the inner surface of a glass container attract to the glass, causing the curving of the water surface (or the surface of any other liquid).

In laboratory work, when reading a graduated cylinder or a burett, follow these rules regarding the meniscus:

- Level the line of your sight with the bottom of the meniscus
- Read the scale where it lines with the bottom of the meniscus (e.g., the volume is 26.0 ml in Figure 12.3)

OSMOSIS AND REVERSE OSMOSIS

Osmosis is a process in which solutions of different concentration are separated through a semipermeable membrane. **Semipermeable membrane** is a membrane with microscopic pores that is permeable to the solvent (mostly water) but nearly impermeable to the solute (solution).

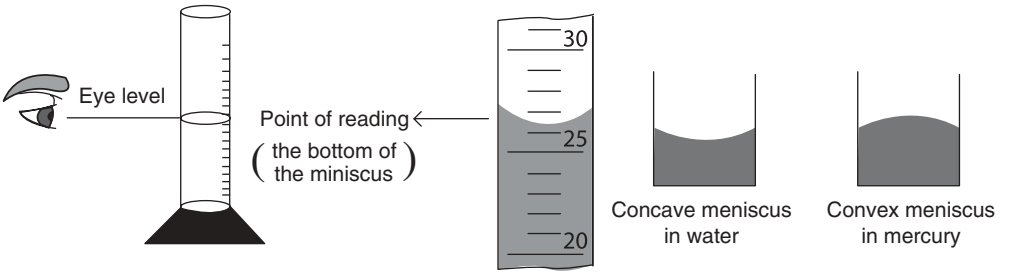


Figure 12.3 Reading a laboratory cylinder or buret

For example, water can pass through a semipermeable membrane but molecules larger than water (e.g., salt) cannot. Several polymers have much higher permeability for water than for dissolved salts. Cellulose acetate mixed with acetyl is one of the best semipermeable membranes.

In the osmosis process, water molecules move from the side where the concentration of water molecules is higher to the side where the concentration of water molecules is lower. For example, if a salt solution is separated from water by osmosis process, the rate at which water molecules pass through the membrane from the freshwater side into the saltwater side is higher than the rate at which they pass from the saltwater side into the freshwater side. The reason is that there are more water molecules in the freshwater side than in the saltwater side. The result of water molecules movement is an increase in the volume of the saltwater side, which causes a buildup in pressure, known as **osmotic pressure**. This pressure forces some water molecules to move across the membrane to the freshwater side. The tendency of molecules to move decreases as the concentration difference decreases. Eventually, the rate at which water molecules move from either side becomes the same and the system reaches **equilibrium**. If an external pressure is now applied to the saltwater side, even more water molecules are forced to move to the freshwater side; this is the process known as **reverse osmosis**.

Reverse osmosis is the opposite technique to osmosis process. Contrary to osmosis, in reverse osmosis water molecules are forced to move through the membrane from the side where the concentration of water molecules is lower to the side where the concentration of water molecules is higher. This is achieved by applying an external pressure greater than the osmotic pressure on the side where the concentration of water molecules is low (saltwater side).

The reverse-osmosis process is used in some labs and households to prepare **deionized water** (DI water). Industrially, it is used in **desalination** of seawater and in some other industries. It can separate up to 98% of the dissolved solids in the water. Desalination is important because of a shortage of drinking water (about 98% of world's water is salty). It currently supplies only about 0.1% of drinking water because of its high operational costs due to high-energy demand.

NOTE

Now that you have studied the basic definitions and equations relevant to chemistry, you can proceed to the second important section of this chapter, the Basics of Math and Statistics.

CHAPTER 12

SECTION 2

BASICS OF MATHEMATICS AND STATISTICS

SUBSECTIONS

- Significant Figures and Rounding Numbers
- Exponents
- Square Roots
- Log
- Antilog
- Polynomial and Quadratic Equations
- Accuracy and Precision of Experimental Results
- Averages
- Standard Deviation
- Coefficient of Variation
- Charts
- Regression Equation

ABOUT THIS SECTION **This section** provides the basics of math and statistics. Statistical topics such as accuracy, average, weighted average, standard deviation, and regression equation are included. The **regression equation** gives you the best solution for some calculations. You will also learn how the **computer** can help you find regression equations quickly and more accurately.

Sugar technologists and sugar chemists often use certain mathematical and statistical operations in their routine calculations, so a review of these operations is included in this section. When you are comfortable with these basic operations, you will be in a good position to solve chemistry problems related to sugar technology.

Beet-Sugar Handbook, by Mosen Asadi
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SIGNIFICANT FIGURES AND ROUNDING NUMBERS

Significant figures are the number of meaningful **digits** (the numeral values 0 to 9 that make up all the numbers) used to express a value. Usually they are the number of digits known with complete certainty plus one additional **estimated digit** (uncertain digit).

For example, assume that you are asked to determine the density of a thin-juice sample. To find the density (*d*), you need to measure the mass and volume of the sample and divide the mass by the volume. Assume that the mass of the sample, measured by an analytical balance, is 52.0022 g. This means that the mass of the sample represents six significant figures, or to the nearest tenth of a milligram (mg). Assume that the volume of the sample, measured by a graduated cylinder, is 49.0 milliliter (ml). This means that the volume was measured to the nearest tenth ml and that its exact value lies between 48.95 and 49.05 ml. To find the density of the sample, you usually use your calculator to divide 52.0022 by 49.0. The displayed result is 1.0612694 (with 8 significant figures, or 7 decimal places). Here, the question is: How do you report the result? The calculator knows nothing about **certainty**, but you know that even if you had an analytical balance and a graduated cylinder accurate to the seventh decimal place (there are not any), trying to measure a sample to that accuracy would be impossible. That's why scientists have determined the rule of **rounding numbers**.

The goal of rounding numbers (e.g., 1.0612694) is to leave the numbers with realistic significant figures. As stated previously, the number of significant figures is the number of digits known with *complete certainty plus one*. Because the least accurate number in this measurement is 49.0 with three significant figures, the answer should be rounded to *four significant figures*. Therefore, the density of the sample has to be reported as 1.061 g/ml (or g/cm^3).

Now, you need to know how to determine the number of significant figures in a set of digits. The digits 1 through 9 are significant figures. The digit 0 has some abnormality (irregularity); in other words, we have to know how to treat 0. The following examples clarify the abnormality of 0 in different situations:

Zeros after a nonzero digit are significant:

500	3 significant figures
50.8	3 significant figures

Zeros between two nonzero digits are significant:

508	3 significant figures
3009	4 significant figures

Zeros to the right of a decimal are significant:

114.0	4 significant figures
11.40	4 significant figures
0.**1100**	4 significant figures
0.0**1140**	4 significant figures

Zeros to the left of a nonzero digit are not significant:

0.0**45**	2 significant figures
0.000**408**	3 significant figures

You must follow certain rules when rounding. Following are examples of rounding figures to the *nearest 0.01* digit:

82.3436	rounds to	82.34
82.3466	rounds to	82.35
82.3450	rounds to	82.35
82.3455	rounds to	82.35

NOTES

- **Rounding should be performed after all other calculations have been made: 12.3 × 123.4 × 0.012 = 18.21384 rounds to 18 because 0.012 has only two significant figures. In the same way, 123.456/48 = 2.572 rounds to 2.6**
- **To avoid mistakes, some scientific calculators have a FIX button that allows you to set the number of digits displayed.**

EXPONENTS

Exponents (powers) are numbers with the power of 10. The following is a list of powers of 10:

$$10^0 = 1$$

$$10^1 = 10$$

$$10^2 = 10 \times 10 = 100$$

$$10^3 = 10 \times 10 \times 10 = 1000$$

$$10^4 = 10 \times 10 \times 10 \times 10 = 10000$$

$$10^{-1} = \frac{1}{10^1} = 0.1$$

$$10^{-2} = \frac{1}{10^2} = \frac{1}{100} = 0.01$$

$$10^{-3} = \frac{1}{10^3} = \frac{1}{1000} = 0.001$$

$$10^{-4} = \frac{1}{10^4} = \frac{1}{10000} = 0.0001$$

Any number may be expressed as a power of 10, or as the product of two numbers, one of which is a power of 10. For example,

$$100 = 1 \times 10^2$$

$$123 = 1.23 \times 10^2$$

$$0.123 = 1.23 \times 10^{-1}$$

$$3000 = 3 \times 10^3$$

$$1234 = 1.234 \times 10^{-3}$$

$$0.00001234 = 1.234 \times 10^{-5}$$

Very large or very small numbers are usually expressed using exponential notation, which is more convenient. For example,

$$12300 = 1.23 \times 10^4$$

$$123000 = 1.23 \times 10^5$$

$$0.1230 = 1.23 \times 10^{-1}$$

$$0.0123 = 1.23 \times 10^{-2}$$

$$0.00123 = 1.23 \times 10^{-3}$$

When two exponentials are multiplied, the exponents are added:

$$(1.23 \times 10^3)(1.0 \times 10^{-1}) = 1.23 \times 10^{3-1} = 1.2 \times 10^2 \qquad (1.23 \text{ rounds to } 1.2)$$

When two exponentials are divided, the exponents are subtracted:

$$\frac{(6.6 \times 10^4)}{(2.0 \times 10)} = 3.3 \times 10^{4-1} = 3.3 \times 10^3$$

In multiplication, exponents of like bases are added:

$$10^3 \times 10 = 10^{3+1} = 10^4$$

$$10^{-4} \times 10^2 = 10^{-4+2} = 10^{-2}$$

$$(2 \times 10^{-3})(3 \times 10^5) = 6 \times 10^{-3+5} = 6 \times 10^2$$

In division, exponents of like bases are subtracted:

$$\frac{10^3}{10^2} = 10^{3-2} = 10$$

$$\frac{10}{10^4} = 10^{1-4} = 10^{-3}$$

Transferring from the numerator to the denominator of a fraction is given as follows:

$$10^2 = \frac{1}{10^{-2}}$$

$$4 \times 10^3 = \frac{4}{10^{-3}}$$

$$4a \times 10^{-3} = \frac{4a}{10^3}$$

The following are a few examples of fractional exponents:

$$(10^3)^2 = 10^{3\times2} = 10^6$$

$$(4^{-3})^{-2} = 4^{-3\times2} = 4^{-6}$$

$$(16^{-3})^{-2} = 16^{-3\times-2} = 16^6$$

NOTES

- **To find a number with a power on a calculator, enter the number, press the X^y key, enter the value of the power, and then click the = key.**
- **To find a number with a power on a computer using Excel, click the function key (f_x), find the POWER function, and click it. When asked, enter the number and the power.**

SQUARE ROOTS

Following are examples of roots:

$$\sqrt{10^3} = 10^{\frac{3}{2}} = 10^{1.5} = 31.6$$

$$\sqrt{2.5 \times 10^{-5}} = \sqrt{25 \times 10^{-6}} = \sqrt{25} \times \sqrt{10^{-6}} = 5 \times 10^{-3} = 0.005$$

NOTES

- **To find the root on a calculator, enter the number and press the √ key.**
- **To find the root on a computer using Excel, click the function key (f_x), find the SQRT function, and click it. When asked, enter the number and the square root.**

LOG

A **log** (logarithm) is the inverse of the exponential operation. Earlier, you learned that

$$1 = 10^0$$

$$10 = 10^1$$

$$100 = 10^2$$

The log to the base of 10 (often written **log** and called common log) of 1, 10, and 100 are as follows:

$$\log 1 = 0$$

$$\log 10 = 1$$

$$\log 100 = 2$$

In general, if $y = a^x$, we define $x = \log_a y$ (read "log base a of y"). For example,

$$100 = 10^2$$

$$10 = 10^1$$

$$1 = 10^0$$

$$0.1 = 10^{-1}$$

$$0.01 = 10^{-2}$$

The common log of a number is the power to which 10 must be taken to produce that number. Because $100 = 10^2$, so log 100 = 2 or log 0.1 = –1.

NOTES

- **Log is the same as $\log_{10}$ (log to the base of 10).**
- **The exponent of 10 for a number between 1 and 10 will be between 0 and 1. For example, $2 = 10^{0.30103}$ and log 2 = 0.30103.**
- **The exponent of 10 for a number between 10 and 100 will be between 1 and 2. For example, $20 = 10^{1.30103}$ and log 20 = 1.30103.**
- **The exponent of 10 for a number between 100 and 1000 will be between 2 and 3. For example, $222 = 10^{2.346353}$ and log 222 = 2.346353.**
- **General properties of the common logs are as follows:**

 The log of a number greater than 1 is positive. For example, log 1.3 = 0.11

 The log of a number lesser than 1 is negative. For example, log 0.3 = –0.52.

 Negative numbers do not have logs.
- **To find the log on a calculator, enter the number and press the log key.**
- **To find the log on a computer using Excel, click the function key (f_x), find the log 10 function, and click it. When asked, enter the number and the log.**
- **Logs are manipulated according to the rules for exponents:**

$$\log ab = \log a + \log b$$

For division,

$$\log \frac{a}{b} = \log a - \log b$$

$$\log a^n = n \log a$$

- **The log was widely used to solve multiplications and divisions before the electronic calculator and computer were invented.**
- **The natural log (ln) is another form of log. This is a log to the base of $e = 2.71828$. The natural log is used in chemistry and physics for solving some differential equations. The natural log is solved according to the following general notation:**

$$\ln x = y \quad x = 2.71828^y$$

- **The following formula can be used to convert a common log to a natural log, and vice versa:**

$$\ln x = 2.303 \log x$$

- **To find Ln on an electronic calculator, enter the number and press the ln key.**

ANTILOG

An **antilog** (antilogarithm) is the same as an exponentiation, meaning raising the base to a power equal to the log. The antilog of 2 is the number whose log is 2. The antilog of any number N can be solved as $N = 10^N$. For example, the antilog of 2 is $10^2 = 100$.

$$\log T = 1.5$$

$$T = 10^{1.5} = 31.6$$

NOTES

- **A calculator can calculate the antilog of numbers. There are different methods for carrying out the antilog operation on various calculators. The most common is to enter the value and press the 10^x key. For example, the antilog of 1.234 is 17.140.**
- **Other calculators have the INV (inverse) function instead. In this case, enter the value, press INV, and then press the log bottom.**

POLYNOMIAL AND QUADRATIC EQUATIONS

A **polynomial equation** has the following form:

$$y = a_1x + a_2x^2 + a_3x^3 + \ldots + a_nx^n$$

A **quadratic equation** is a polynomial in which the highest power of x is 2. Therefore, the quadratic equation has the general form of

$$ax^2 + bx + c = 0$$

In this equation, a, b, and c are constant. For example,

$$x^2 + 2x - 3 = 0$$

In this example, $a = 1$, $b = 2$, and $c = -3$

The general solution to the quadratic equation is given as

$$x = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a}$$

Note that a quadratic equation has two roots (positive and negative). The preceding example can be solved as

$$x = \frac{-2 \pm \sqrt{2^2 - 4 \times 1 \times -3}}{2 \times 1} = \frac{-2 \pm \sqrt{4 + 12}}{2} = \frac{-2 \pm \sqrt{16}}{2} = \frac{-2 \pm 4}{2}$$

$$x_1 = \frac{-2 + 4}{2} = 1$$

$$x_2 = \frac{-2 - 4}{2} = -3$$

Polynomial equations, in sugar technology, are used often to solve the **solubility** (see the Glossary) of the substances in a **saturated solution** (see the Glossary).

For example, the relationship between the solubility of sucrose and the temperature of the solution can be expressed by a polynomial equation (refer to Section 16 of Chapter 3). Therefore, solubility of sucrose (S) at T°C can be expressed as

$$S = 64.447 + 0.08222T + 1.6169 \times 10^{-3}T^2 - 1.558 \times 10^{-6}T^3 - 4.63 \times 10^{-8}T^4\% \qquad (12.24)$$

For example, the amount of sucrose that can be dissolved in water at 20°C is

$$S = 64.447 + 0.08222 \times 20 + 1.6169 \times 10^{-3} \times 20^2 - 1.558 \times 10^{-6} \times 20^3 - 4.63 \times 10^{-8} \times 20^4$$

$$S = 66.74\%$$

ACCURACY AND PRECISION OF EXPERIMENTAL RESULTS

Physical sciences (e.g., chemistry and physics) are based on the results of **measurements**. **Statistics** help us to better organize the results of any measurement for reaching a better conclusion. For example, in a sugar laboratory we run several daily tests to find out the sugar (sucrose) content in sugarbeet. After the test results are averaged, we want to know two important concepts:

- **Accuracy:** How good our measuring device is in getting a correct result
- **Precision:** How close the values are to each other

The accuracy and precision of a test depend on the following:

- Familiarity of the analyst with the test
- Accuracy of the calibration of the instrument used in the measurement
- Accuracy of the procedure used in the measurement

For reaching a conclusion, we must be able to estimate the size of **error** (uncertainty) involved with the test. A measurement always has some degree of error. The two kinds of errors are

- Systematic error
- Random error

It is possible to eliminate **systematic error**, but **random errors** (errors by chance) can only be minimized. A systematic error occurs in the same direction each time; it is either always high or always low. For example, the incorrect calibration of a polarimeter creates systematic error.

The **accuracy** of a measurement refers to how close the measurement is to the **true value** (accepted theoretical value). An error relating to accuracy is called a **percent error**, which is calculated as follows:

$$\%\ \text{Error} = \frac{\text{Accepted value} - \text{Experimental value}}{\text{Accepted value}} \times 100 \qquad (12.25)$$

EXAMPLE 36

Given:

Density of water at 4°C was measured to be	1.03 g/ml
Accepted density for water at 4°C is	1.00 g/ml

Calculate:
Percent error

$$\%\ \text{Error} = \frac{1.00 - 1.03}{1.00} \times 100 = \pm 3$$

NOTES

- **The error limit (indicated by ±) represents the uncertainty of the measurement result. Mathematically, the error limit is the number that gives the high and low range values. In the preceding example, the error limit is +3% and –3%, meaning that we can report the density as 1.03 ± 0.03 g/ml.**
- **The confidence range is the range of values within experimental error. In the preceding example, the confidence range is 100 – (±3) = ±97%.**

Precision (reproducibility) refers to how close the measurement values are to each other. In other words, precision refers to the degree of agreement among several measurements of the same quantity. For example, if two analysts weigh the same object on the same balance, most likely they will not obtain the same result because a measurement always has some degree of error (experimental uncertainties). In this particular case, for example, the current air may change its direction and affect the balance differently when each analyst weighs the same object.

To understand how accuracy and precision compare, refer to the following note.

NOTE

- **If a measuring device is not properly calibrated, it may give high precision, but none of the measurements is accurate (low accuracy). However, a properly calibrated measuring device is both accurate and precise. Therefore, a systematic error can be avoided through routine calibration (comparing the measuring device with a known standard). But random errors can only be minimized.**

In chemistry, **precision** is defined as the degree of **distribution** (dispersion) of the results. The most common way to specify precision is by the **standard deviation** (*SD*) or **coefficient of variation** (*CV*), which are explained later, after you learn about **averages**.

AVERAGES

An **average** is the one value that best represents the result of a measurement. The average can be expressed in three ways:

- Mean
- Median
- Weighted average

Mean

The **mean** (ordinary average) of a data set is the sum of the values (entries) divided by the number values. To find the mean ($\bar{x}$) of a data set, the following formula is used:

$$\bar{x} = \frac{\sum x}{n} \tag{12.26}$$

Here, $\bar{x}$ (read x bar) is the mean (average) of the values, sigma (Σ) indicates a summation, x is each individual value (data), and n is the total number of values. Put simply, to find the mean, follow three steps:

1. List the values in one column
2. Compute the sum (total) of values
3. Divide the sum by the number of values

Median

The **median** (midpoint) is the value that lies in the middle of the results. Therefore, 50% (one-half) of the values are above the median, and 50% are below the median.

There is no formula for calculating the median. To find the median, the procedure is even simpler:

1. List the values in order (from highest to lowest or vice versa).
2. If the number of values is odd, the median is the middle value. If the number of values is even, the median is the average of the two middle values.

NOTE

Mean aperture (*MA*) or mean opening is used to determine the crystal size distribution (CSD) of granulated-refined (GR) sugar. *MA* is defined as the size of a sieve for which 50% of the sugar is retained and 50% passes through. Although this definition is the same as that for the median, sugar technology still uses the traditional term mean aperture.

Weighted Average

The **weighted average** (weighted mean) is the average of a data set in which values have different weights (greatness). The weighted average is used when the data set contains values that have a **greater effect** (weight) on the average than do other values. The weighted average is given by

$$\overline{x}_w = \frac{\sum (x.w)}{\sum w} \tag{12.27}$$

$\overline{x}_w$	Weighted average
w	Weight of each value x

To find the weighted average, follow these steps:

1. List the values in one column
2. List the weights in another column
3. Find sum of the weights (Σw)
4. Multiply each value (x) by its weight (w) and find their sum ($\Sigma x.w$)
5. Divide the sum determined in step 4 ($\Sigma x.w$) by the sum determined in step 3 (Σw)

The **mean**, **median,** and **weighted average** are all averages, but each works better in particular situations. The following are helpful guidelines for selecting which average to use:

- Use the **mean** when the data do not contain extreme values.
- Use the **median** when the data contain **extreme values** (out-of-line values).
- Use the **weighted average** when the data contain values that have a **greater effect** (weight) on the average than do other values.

Consider the average selectivity in the context of the following example:

EXAMPLE 37

Given:

In a beet-sugar factory with a beet-slice capacity of 300 t per hour, the diffusion juice was tested for purity every hour, and the amount of beets processed during the hours of each shift were recorded as follows:

First Shift			Second Shift			Third Shift		
Time	*Sliced*	*Purity*	*Time*	*Sliced*	*Purity*	*Time*	*Sliced*	*Purity*
7.00	294	89.6	3.00	294	89.0	11:00	295	86.6
8:00	287	90.2	4:00	302	90.1	12:00	288	86.2
9:00	304	88.8	5:00	289	89.6	1:00	Shutdown	
10:00	297	89.3	6:00	291	89.1	2:00	Shutdown	
11:00	289	90.5	7:00	296	90.4	3:00	**28**	88.5
12:00	287	89.2	8:00	299	89.5	4:00	**77**	88.9
1.00	302	90.1	9:00	293	**86.3**	5:00	**95**	89.1
2.00	296	89.7	10:00	292	**85.1**	6:00	**122**	89.3

Determine:

The best way of averaging to report the results of the purity of diffusion juice for the first, second, and third shifts

The **first-shift** data indicate that the factory operated normally without any problems. None of the data (the amount of processed beets and the purity of the diffusion juice) during the first shift were out of line (extreme). The amount of beets sliced per hour is close to the nominal capacity of the factory (the minimum slice rate during the first shift is 285 t per hour and the maximum is 304 t per hour), and the purity values are within normal range (minimum is 89.3 and maximum is 90.5). Therefore, the **mean** is the best choice.

$$\bar{x} = \frac{\sum x}{n} = \frac{717.4}{8} = 89.7$$

The **second-shift** data on purity contain two extreme values (86.3 and 85.1). This indicates that a problem started to happen during the last hour of this shift. Due to the extreme values, the **median** is the best choice. To find the median, the values should be listed in order:

Purity (in order)
90.4
90.1
89.6
89.5
89.1
89.0
86.3
85.1

Because the number of values is even, the median is the average between the two middle values:

$$\text{Median} = \frac{89.5 + 89.1}{2} = 89.4$$

The **third-shift** data contain abnormal values due to operational problems in the diffuser. As seen from the data, the factory had to shut down for two hours to correct the problem. Here, the values for 11:00 to 1:00 have a **greater effect** on the average because during those hours the factory was in full operation and sliced more sugarbeet than during the rest of the shift. Therefore, in this situation a **weighted average** is more correct than the mean or median:

w	x	$x.w$
295	86.6	25547
288	86.2	24826
28	88.5	2478
77	88.9	6845
95	89.1	8465
122	89.3	10895

$\Sigma w = 905 \quad \Sigma(x.w) = 79056$

$$\overline{x_W} = \frac{\Sigma(x.w)}{\Sigma w} = \frac{79056}{905} = 87.4$$

STANDARD DEVIATION

The **standard deviation** (*SD*) is a way of describing the **precision** of the result of a set of measurements. Statistically, *SD* is defined as the average deviation, or distribution (dispersion), of all values of a data set. The simplest way to estimate *SD* is to deduct the largest value in the set from the smallest value in the set and divide the result by 2. However, the most accurate way to calculate *SD* is by the following formula:

$$SD = \sqrt{\frac{\Sigma(x - \bar{x})^2}{n - 1}} \tag{12.28}$$

$\bar{x}$ Average (either mean, median, or weighted average)
n Total number of measurements

NOTES

- **The *smaller* the standard deviation, the more precise will be the measurements. Therefore, the smaller *SD* is desired.**
- **Minus signs are ignored when we calculate $x - \bar{x}$.**
- **Excel has a feature that can calculate the standard deviation for paired values, explained later.**

COEFFICIENT OF VARIATION

The **coefficient of variation** (*CV*) is another way of describing the **precision** of the result of a set of measurements. Statistically, the *CV* is defined as the relative deviation of the values of a data set. *CV* is given as

$$CV = \frac{SD}{\bar{x}} \times 100 \tag{12.29}$$

NOTE

When running a test by two different methods, the method that has the smaller *SD* or *CV* has better precision (a high *SD* or *CV* value characterizes a wide and poor distribution).

EXAMPLE 38

Given:

The specification for a commercial 1 kg bag of sugar that meets quality control (QC) of the packaging operation of a sugar factory is that each bag not contain less than 1000 and more than 1015 g of sugar. Assume that the QC of several bags gave the following results: 1009, 998, 1003, 1017, and 1019.

Calculate:

How can we use these results to decide if the weights meet the specification? In other words, we want to calculate the following:

- Mean
- Median
- Standard deviation
- Coefficient of variation

The mean of the test results is

$$\bar{x} = \frac{\sum x}{n} = \frac{1009 + 998 + 1017 + 1019}{5} = 1010\,\text{g}$$

The median is the middle value (1009) because the number of values is odd.

The standard deviation (*SD*) is

$$SD = \sqrt{\frac{\sum (x - \bar{x})^2}{n - 1}}$$

$$SD = \left[\frac{(1009 - 1010)^2 + (998 - 1010)^2 + (1003 - 1010)^2 + (1017 - 1010)^2 + (1019 - 1010)^2}{5 - 1}\right]^{1/2}$$

$$SD = \left(\frac{1 + 144 + 49 + 49 + 81}{4}\right)^{1/2} = 81^{1/2} = \pm 9$$

$$CV = \frac{SD}{\bar{x}} \times 100 = \frac{9}{1010} \times 100 = 0.9\%$$

- The **mean** value (1010) expresses the average of the measurements indicating that the specifications were met.
- The **median** value (1009) indicates that in this experiment 50% of the time the weights of the packages fall above 1009 g, and 50% of the time they fall below 1009 g.
- The **standard deviation** (9) helps us express the precision of this experiment. We can say that the amount of sugar in the typical package is 1010 g with a sample standard deviation of 9 g. Statistically, this means that any additional control has a 91% probability (91 chances out of 100) of being between 1019 g (1010 + 9) and 1001 g (1010 – 9).
- The **coefficient of variation** (0.9%) is another way of expressing precision. When the *CV* is found to be a small value, it indicates a **high confidence**.
- Note that Excel can also calculate these data as follows:
 1. Click **fx** on the **toolbar** (the set of icons below the menus).
 2. In the Function category, click Statistical.

CHARTS

Charts (graphs) display the values of a measurement for better understanding and evaluating the data. Charting is a valuable tool in chemistry. For example, consider a chart in which the density (***d***) of pure sucrose solutions is plotted as a function of their dry substance (***DS***). This chart can be constructed by marking the density for each dry substance and drawing a curve through the points.

Using a computer is the easiest way to create a chart. (The charts in this book were created using **Microsoft Excel**.) To create a chart in Excel, you must first create a table. Then use Excel's **Chart Wizard** to draw a chart for yourself as follows:

1. Click **Chart Wizard** on the **toolbar** (the set of icons underneath the menus).
2. Choose the **chart type** (e.g., bar, line, or **xy**) and then click **Next**.
3. Choose the **chart source** by clicking the table, highlighting the data you want in the chart, and then clicking **Next**.
4. Chose the **chart option**, and then click **Next**.
5. Choose the **chart location**, and then click **Finish**.

Figure 12.4 shows the relationships between the variables *DS* and *d* of sucrose solutions for five sets of data (densities of the solutions with 0%, 20%, 40%, 60%, and 80% *DS*).

REGRESSION EQUATION

A **regression equation** defines mathematically the **relationship** (correlation) between two or more variables. The regression equation and **regression line** (the line drawn based on the values in the regression equation) are used in many mathematical applications. Regression equations are particularly helpful today because they can change the data of some tables to a computer-workable form.

One common application is when two variables have a **proportional** (linear) **relationship** with each other. For example, the dry substance (*DS*) of a sugar solution has a linear relationship with its density (*d*) with a high **degree of correlation**. Or the percent transmittance (*T*)

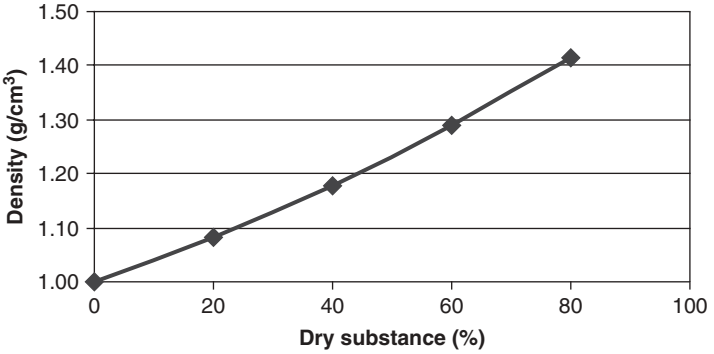


Figure 12.4 Relation between dry substance and the density of sucrose solutions

of a sugar solution is proportional to the color value (C) of the solution. When two variables indicate a high degree of relationship (correlation), we can determine the value of one variable by substituting the value of other variable into the regression equation. Therefore, we are no longer dependent on the table that indicates the data related to these two variables (e.g., Table A.1 given in the Appendix).

To determine a regression equation, first you key in a chart of values in a computer (on an Excel spreadsheet) as explained in the previous subsection. In this situation, a scatter (xy) chart is one of the best ways to graph the data. Then, Excel can determine the regression equations and the R-squared value as follows:

1. Draw your chart as previously explained
2. Click the trendline for which you want to display the equation and R-squared
3. On the **Chart** menu, click **Add Trendline**
4. On the Options tab, select **Display Equation** and **Display R-squared**

The **R-squared** (R^2) value indicates the degree of linearity (proportionality) between the variables (*the closer the value of R^2 to 1, the stronger is the degree of linearity between the variables*). In other words, when R^2 is equal to 1, the two variables have a perfect (absolute) linearity.

To determine the relationship between two variables (DS and d) and their proportionality, we use the data in Table A.1 of the Appendix. We determine the regression equation and the degree of linearity (R-squared value) for DS and d variables by following the preceding steps. The result is the graph shown in Figure 12.5, based on the equation and the R-squared (R^2) values obtained for these variables.

The correlation (relationship) between two variables, represented by a regression equation, may be **linear** (straight line) or **nonlinear** with positive or negative **correlation**. For example, the relationship between the variables in the preceding case is a linear pattern with a **positive correlation** (when one variable increases, the other increases too). When we have a linear relation between variables, we say that y is a linear function of x. A linear relationship can be expressed by a linear equation:

$$y = ax + b$$

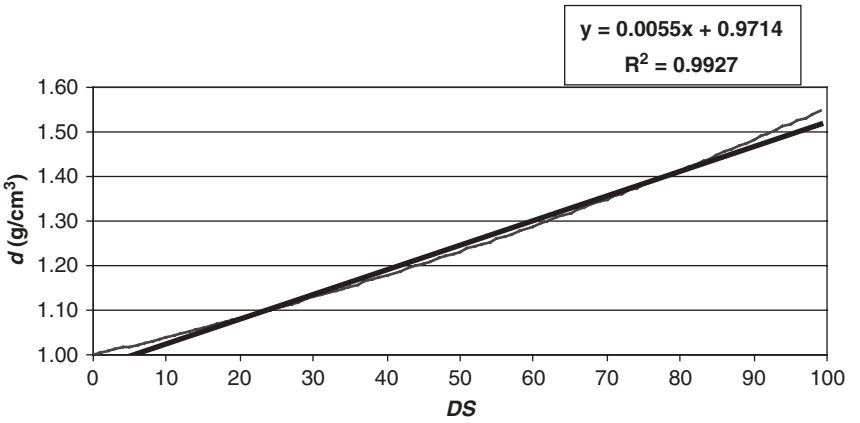


Figure 12.5 Graph of a regression equation created by using a computer

y	Dependent variable (y is the function of x)
x	Independent variable
a	Slope of the line
b	Intercept of the line (point of intersection)

Note that the intercept occurs when $x = 0$, and then

$$y = a \times 0 + b = b$$

The slope of a straight line is defined as the ratio of the rate of change in y to that in x:

$$a = \text{Slope} = \frac{\Delta y}{\Delta x}$$

In the *DS*-density equation (the regression equation whose variables are *DS* and *d*) found by computer (see Figure 12.5), the value of slope a is equal to 0.0055, and the value of the intercept b is equal to 0.9714. Therefore, the equation can be expressed in the following ways:

$$y = 0.0055x + 0.9714 \tag{12.30}$$

$$d = 0.0055 \times DS + 0.9714 \tag{12.31}$$

The values of x (in this case, *DS* of the solution) are independent because they are known, but the y values (density of the solution) are dependent because they are unknown. After finding the regression equation for variables, we can choose any real value of *DS* and produce a value for *d*. For example, the density of a sugar solution with 15.0% *DS* is equal to:

$$d = 0.0055 \times 15.0 + 0.9714 = 1.054\,\text{g/ml}$$

By using the *DS*-density equation, we are no longer **dependent** on their relationship table (Table A.1 of the Appendix). This equation is used in the Spectrophotometer subsection of Section 2 of Chapter 11 to calculate the color of sugar solutions.

END-OF-THE-CHAPTER NOTE

- **Chapter 12** (in only about 50 pages) gave you basic concepts of chemistry, math, and statistics to enable you to understand the technology and chemistry of sugar production and all examples given in different chapters of this book.

APPENDIX

SECTIONS

- Use of Computer on Operation Reports
- Units of Measurements
- Conversion Factors
- Nonsugar-Elimination Formula
- Spider Diagram
- Tables

ABOUT THIS APPENDIX **This appendix** is a useful guide to using a computer for tasks such as preparing factory operation reports. You will also find out how to set up **regression equations** of variables so that you can make calculations on the computer independent of some tables. Furthermore, the appendix provides 18 tables used in sugar-technology calculations.

USE A COMPUTER FOR OPERATION REPORTS

Several software programs can be used for calculations. This book uses **Microsoft Excel**, which can perform basic mathematical calculations as well as many statistical calculations, such as squared, square root, R-squared, logs (natural or base 10), standard deviation, regression equation, and correlation coefficient (coefficient of variation). The program also presents data graphically using charts (graphs). In addition, you can automate Excel even further through the use of **macros**.

Beet-Sugar Handbook, by Mosen Asadi
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Macros

Macros reduce a series of actions to a single keystroke, menu choice, or button push. Using macros reduces what otherwise would take hours of manual effort. By automating repetitive actions, we can dramatically speed up our work. Macro instructions are written in Visual Basic, which is used in various Microsoft applications to automate actions. Visual Basic code can be typed into a module in Excel or created automatically using the macro recorder. For example, the following code selects a specific cell, enters a name, and then applies formatting to the cell (Dale Anderson 2002):

```
Sub SampleMacro()
Range("B3").Select
ActiveCell.Formula = "Lab"
With Selection.Font
Name = "Book"
FontStyle = "Bold Italic"
Size = 18
ColorIndex = 5
End With
End Sub
```

Collecting Lab Data

Excel can be used as an efficient tool to collect data. Figure A.1 shows a portion of a spreadsheet used to collect lab data. This type of spreadsheet is simple to create. Where it gets difficult is when we need a spreadsheet like this for every day of a campaign. Duplicating several hundred identical spreadsheets would be tedious; this is where macros become a big help.

Duplicating Documents

A macro can automate tedious tasks. The following macro makes simple work out of creating hundreds of identical documents:

```
Sub DuplicateSheets()
    CurrentLocation = ActiveWorkbook.Path
    Application.ScreenUpdating = False
    Application.DisplayStatusBar = True
    For Counter = 1 To 360
        Application.StatusBar = "Creating Sheet " & Counter
        Sheets(1).Activate
        ActiveSheet.Unprotect
        Range("B1").Value = Counter
        ActiveSheet.Protect
        ActiveWorkbook.SaveAs CurrentLocation & "\Day" & _
            Format(Counter, "000") & ".xls"
    Next Counter
    ActiveWorkbook.Close
    Application.StatusBar = False
End Sub
```

	Thin juice				Thick juice		Limesalts				Limecake			CO2	Pulp & Pellets				
	%	%	%		%		Thin juice			Mol-	%	%	%	%	% Moisture				%Sug.
	RDS	Pol	Purity	Color	*RDS*	Color	Hard	S. Ash	Soft	asses	Sugar	CaO	Moist.	i n	Wet	Press	Dry	Pellets	Pellets
7am																			
8																			
9																			
10																			
11																			
12pm																			
1																			
2																			
3																			
4																			

Figure A.1 A laboratory report sheet

This macro takes an existing document (similar to the one given in Figure A.1) and creates 360 duplicates. The files are identical except that each has a unique number placed in cell B1 to identify the day and each file has a unique name.

NOTE

- **Programmable logic control (PLC) or simply programmable controller is a system that is programmed to automatically control equipment. A PLC contains a central processing unit (CPU), input and output modules, and a programming device.**

UNITS OF MEASUREMENTS

Any measurement of a dimension includes a **number** and a **unit**. Two unit systems are used in the world:

- Metric system
- English system

Because the United States and two other countries (Liberia and Myanmar) are the only countries in the world that still use the English system, this system is often called the **United States Customary system** (USC system).

NOTES

- **Thomas Jefferson and several other American presidents tried without success to change the unit system in the United States. Some industries in the United States have already switched to the metric system. The U.S. sugar industry is not one of them.**
- **In the United States, when we deal with factory operation reports, sugar entered in the factory is expressed in short tons, sugar production is in pounds or cwt (hundredweights), molasses production is in short tons, and crystallization products are in cubic feet. This requires several conversions between units, which is inconvenient and can create errors. If we were to use the metric system, all these values would be expressed in metric tons.**
- **In long calculations, it is much easier to do basic calculations in metric units and then convert the end result to the English system.**
- **This book gives metric units, followed by the English unit in parentheses. In Chapters 11 and 12, all units are in the metric system because that is standard in chemistry.**

Two types of units are used:

- Fundamental units
- Derived units

Fundamental units are length, mass, and time. Each fundamental unit has a specific definition. For example, kilogram (**kg**, a unit of mass) is defined as the mass of 1 liter of water at 4°C.

Derived units are combinations of fundamental units. For example, **volume** is a derived unit and is a cubic of length, indicated as $\mathbf{m^3}$ in the metric system and $\mathbf{ft^3}$ in the English system. Other examples of derived units are area and speed.

Metric and English fundamental units are as follows:

Measure	Metric Unit	English Unit
Length	Meter (m)	Foot (ft)
Mass	Gram (g)	Pound (lb)
Time	Second (s)	Second (s)
Temperature	Degree Celsius (°C)	Degree Fahrenheit (°F)
Energy	Calorie (cal)	Calorie (cal)

The **metric** (SI) system is easy to work with because most values are based on a multiple or division of 10. This means that conversion measurements can be multiplied or divided by 10. A meter (**m**) divided into 100 is a centimeter (**cm**). Each centimeter consists of 10 smaller units called millimeters (**mm**). Because the main units are not always convenient (expressing the mass of a filter paper in kg is difficult), the metric system uses **prefixes** to change the size of the unit (e.g., gram to milligram).

Prefix	Symbol	Multiplier
mega	**M**	$1\,000\,000 = 10^6$
kilo	**k**	$1000 = 10^3$
hecto	**h**	$100 = 10^2$
deka	**da**	$10 = 10^1$
deci	**d**	$0.1 = 10^{-1}$
centi	**c**	$0.01 = 10^{-2}$
milli	**m**	$0.001 = 10^{-3}$
micro	**μ**	$0.000\,0001 = 10^{-6}$

For example, a cube that measures 1 meter (m) on each edge has 1 cubic meter (m^3) of volume. And $1\,m^3$ is 10 cubic decimeters (dm^3). Note: In the metric system, a cubic decimeter ($10 \times 10 \times 10 = 1000\,cm^3$) is called a liter (L) and a cubic centimeter (cm^3) is called a milliliter (ml). One liter is slightly larger than a quart (946 ml), which is one of the units used for volume in the USC (English) system.

NOTES

- **The unit of mass is kilogram (kg), but gram (1 g = 1/1000 kg) and millgram (1 g = 1/1000000 kg) are also used.**
- **In the metric system, the unit of force (F) is Newton (N) and the unit of area is square meter (m^2), giving the unit of pressure (force applied over a surface area) as N/m^2 (Newton per square meter), or 1 Pascal (Pa). Because the Pascal unit is so small, multiples of 10^3 Pa (kPa) and 10^5 Pa (1 bar = 100 kPa) are often used. For example, the pressure of automobile tires is 240 kPa.**
- **In the English system, the unit of mass is pound (lb) and the unit of area is square inch ($in.^2$) giving the unit of pressure as pounds per square inch (psi). If 1 lb of force is applied to a surface area of $1\,in^2$, we say that the pressure is 1 psi. For example, the pressure of automobile tires is 35 psi (240 kPa).**

- **Temperature is an independent unit and in chemistry is usually expressed in degrees Celsius (or centigrade, °C), invented in 1741 by Anders Celsius. The centi- prefix in centigrade refers to the 100 equal degrees covering the range between the freezing point of water (0°C) and the boiling point of water at sea level (100°C).**
- **Units must be carried along with the numbers and must be treated the same way as the numbers when quantities have to be multiplied or divided, for example,**

$$5.3\,\text{cm} \times 3.0\,\text{cm}^2 = 15.9\,\text{cm}^{1+2} = 15.9\,\text{cm}^3$$

- **Quantities cannot be added or subtracted directly unless they have the same units, for example,**

$$15.6\,\text{L} + 12.1\,\text{L} = 27.7\,\text{L}$$

CONVERSION FACTORS

Conversion factors (Table A.17, given at the end of the book) are expressions of the relationship between units. For example, 2.2 lb and 1 kg are the same mass (2.2 lb = 1 kg). The conversion factor can be written in two ways:

$$\frac{2.2\,\text{lb}}{1\,\text{kg}} \quad \text{and} \quad \frac{1\,\text{kg}}{2.2\,\text{lb}}$$

For example, the mass of an object that is 12.5 kg is

$$(12.5\,\text{kg})\frac{2.2\,\text{lb}}{1\,\text{kg}} = 27.5\,\text{lb}$$

In calculations, the following general equation can be used:

$$\text{Starting quantity} \times \text{Conversion factor} = \text{Equivalent quantity}$$

A unit of the given quantity is canceled when we use the conversion factor. This type of carrying unit is called **dimensional analysis**. In dimensional analysis, units are canceled, multiplied together, or divided into each other.

The **conversion factor table** is used to convert a unit to another unit. For conversion, follow these steps:

1. Find the equivalent from the conversion-factor table in this appendix.
2. Multiply the quantity, which has to be converted, by the unit factor.

EXAMPLE 1

Given:
A pressure equal to 2 atmospheres (atm)

Convert:
The pressure to kPa (kilo Pascal) and psi (pounds per square inch)

The factor for converting atmosphere to kPa is 101.33; therefore,

$$2 \times 101.33 = 202.66\,\text{kPa}$$

The factor for converting atmosphere to psi is 14.7. Therefore,

$$2 \times 14.7 = 29.4\,\text{psi}$$

Celsius degrees (°C), Fahrenheit degrees (°F), and Kelvin (°K, note that the word **degree** is not used with the Kelvin scale) are three scales for temperature measurements (see Figure A.2). Note also Kelvin has the same size temperature change as a °C, so a change of 1°C is the same as a change of 1 Kelvin. The freezing point temperature of water is 273 K, or °K = °C + 273. To convert Celsius to Fahrenheit or vice versa, use the following equations:

$$°C = \frac{°F - 32}{1.8} \quad \text{Simplified formula is} \quad °C = \frac{°F - 32}{2}$$

$$°F = 1.8°C + 32 \quad \text{Simplified formula is} \quad °F = 2°C + 32$$

NONSUGAR-ELIMINATION FORMULA

Nonsugar elimination (*NSE*) is the percentage (by mass) of nonsugars (*NS*) reduction in those stations involved with nonsugar separation. In other words, *NSE* is the expression of efficiency of the station. *NSE* is determined by comparison of the purity of the feed to a station (P_1) and that of the product leaving the station (P_2).

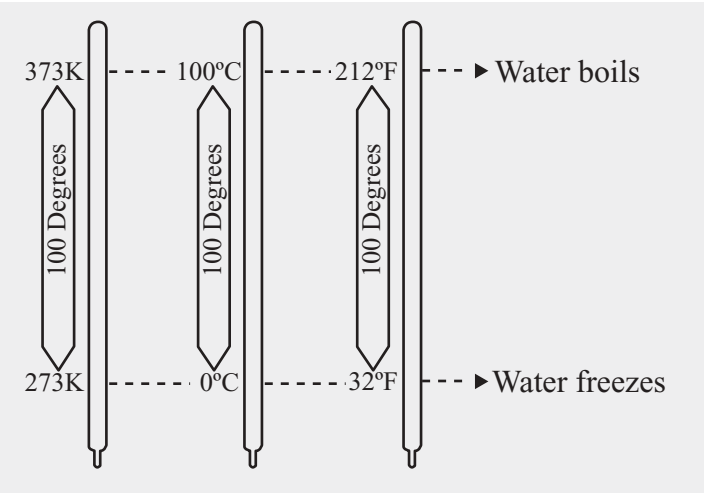


Figure A.2 Comparison of the Celsius, Fahrenheit, and Kelvin scales

Assume that diffusion juice with a purity of P_1 enters the purification station and leaves at a higher purity of P_2 as thin juice. *NSE* is determined by the percentage of nonsugar-to-sugar ratio (*NS*/*S*) between entering and leaving the station. But we do not have a direct measure of nonsugars of either juice, so the purity can be used to calculate *NSE*. Note that the basic assumption for calculations is that the sucrose that enters the station is equal to that which leaves; only nonsugars have been reduced. The following equations represent the percentage of purities at initial and final points:

$$\frac{100 - P_1}{P_1} \quad \text{and} \quad \frac{100 - P_2}{P_2}$$

Consequently, the percentage of difference between two cases is the nonsugar elimination (*NSE*):

$$NSE = \frac{(100 - P_1)/P_1 - (100 - P_2)/P_2}{(100 - P_1)/P_1} \times 100$$

$$NSE = \frac{[(100 - P_1)P_2 - (100 - P_2)P_1]/P_1P_2}{(100 - P_1)/P_1}$$

$$NSE = \frac{(100P_2 - P_1P_2 - 100P_1 + P_1P_2)/P_2}{(100 - P_1)} \times 100 = \frac{100(P_2 - P_1)}{P_2(100 - P_1)} \times 100$$

$$NSE = \frac{(P_2 - P_1)}{P_2(100 - P_1)} \times 10000$$

NOTE

- ***NSE* formula is mainly used to determine the efficiency of the purification and the crystallization station. *NSE* can also be used to express the recovery of the factory based on the purity of the thin juice and molasses (see Section 2 of Chapter 1).**

EXAMPLE 2

Given:

Purity of diffusion juice (P_1)	87.5%
Purity of thin juice (P_2)	90.0%

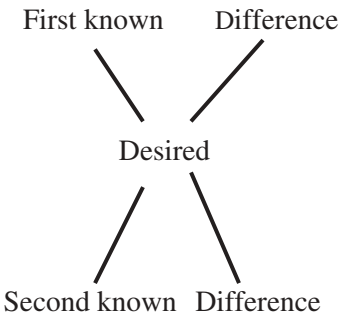
Calculate:

Nonsugar elimination (*NSE*) in the purification station

$$NSE = \frac{(P_2 - P_1)}{P_2(100 - P_1)} \times 10000 = \frac{(90.0 - 87.5)}{90.0(100 - 87.5)} \times 10000 = 22.2\%$$

SPIDER DIAGRAM

The **spider diagram** (Cobenz's diagram) can be used to make a desired concentration from two solutions with known concentration. In factory operations, the spider formula is mostly used in backboiling for the calculation of two syrups to give a mixture of the desired dry substance (*DS*) or purity.



EXAMPLE 3

Given:

Two syrups with 88.0 and 75.0% *DS* are available

Calculate

How much of each has to be mixed to produce 70 m^3 syrup with 83% *DS*



8 parts of 88.0% Syrup and 5 parts of 75.0% Syrup produce 13 parts of 83.0% Syrup.

$$70 \times \frac{8}{13} = 43\,m^3 \text{ of } 88.0\% \text{ Syrup}$$

$$70 \times \frac{5}{13} = 27\,m^3 \text{ of } 75.0\% \text{ Syrup}$$

Total 70 m^3

EXAMPLE 4

Assume that the numbers in the preceding example are **purity** values instead of ***DS***. Suppose that the pan already contains 50 m^3 of massecuite at 88.0% purity, and the sugar boiler wants to bring the purity down to desired value of 83.0% by **backboiling** 75.0% purity syrup. What would be the final volume of the pan?

According to the spider diagram in the previous example, the sugar boiler needs to add (backboil) 5.0 parts of 75.0% purity syrup to the pan, which already has 8 parts of 88.0% purity syrup. Since 50 m^3 is equal to 8 parts of the total parts (13 parts), the final volume of the pan will be

$$50 \times \frac{13.0}{8.0} = 81.3\,\text{m}^3$$

For more information on backboiling calculation, see also Example 5 in Section 17 of Chapter 3.

TABLES

Tables are helpful in routine calculations but cannot be conveniently incorporated into computer-aided calculations. To change some tables to a computer-workable form, you can use **regression equations**. When a high degree of correlation (linearity) between variables in a table exists, we can use a regression equation to make calculations independently of the table.

For example, to be independent of the *DS*-density table (Table A.1 of this appendix), you can use the regression equation of the variables for each individual test and calculate a formula containing the related variables (dry substance [*DS*] and density [*d*]). The regression equation of *DS*-density relation was derived in the Mathematics and Statistics section of Chapter 12 and was used in the Spectrophotometer subsection in Section 2 of Chapter 11 for a fast way to calculate color. In Chapter 12, the regression equation was given as

$$y = 0.0055x + 0.9714 \tag{12.30}$$

$$d = 0.0055 \times DS + 0.9714 \tag{12.31}$$

In using Eq. (12.31), we are no longer **dependent** on the density table. For example, the density of a sugar solution with 15.0% *DS* is equal to

$$d = 0.0055 \times 15.0 + 0.9714 = 1.054\,\text{g/cm}^3$$

Let us compare the result of the density calculated above using the regression equation with data given in Table A.1. For example, the density of a 15.0% sucrose solution given by Table A.1 is 1.061 g/cm^3 and 1.054 using the regression equation. As you can see, the difference is insignificant for practical purposes.

To calculate the density using Eq. (12.31) on an Excel spreadsheet, enter the *DS* value in one cell (e.g., cell A2) and type the density equation in another cell. (Note: You have to type the density equation in the computer as 0.0055*A2+0.9714, where * is the multiplication sign and A2, for example, is the cell in which the density is entered.)

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Table A.1 Density and Baume of Pure Sucrose Solutions (at 20°C)

DS (% m/m)	Density (kg/m^3)	Density (lb/ft^3)	Density (lb/ga)	Baume (degree)	*DS* % (m/m)	Density (kg/m^3)	Density (lb/ft^3)	Density (lb/ga)	Baume (degree)
0	1000.00	62.25	8.32	0.0	11	1044.13	65.00	8.69	6.1
1	1003.89	62.49	8.35	0.6	12	1048.31	65.28	8.72	6.7
2	1007.79	62.73	8.39	1.1	13	1052.52	65.52	8.76	7.2
3	1011.72	62.98	8.42	1.7	14	1056.77	65.79	8.79	7.8
4	1015.67	63.23	8.45	2.2	15	1061.04	66.05	8.83	8.3
5	1017.85	63.47	8.49	2.8	16	1065.34	66.32	8.87	8.9
6	1023.66	63.72	8.52	3.4	17	1069.68	66.59	8.90	9.5
7	1027.70	63.97	8.55	3.9	18	1074.04	66.86	8.94	10.0
8	1031.76	64.23	8.59	4.5	19	1078.44	67.14	8.98	10.6
9	1035.86	64.48	8.62	5.0	20	1082.87	67.41	9.01	11.1
10	1039.98	64.74	8.66	5.6	21	1087.33	67.69	9.05	11.7

Table A.1 Continued

DS (% m/m)	Density (kg/m^3)	Density (lb/ft^3)	Density (lb/ga)	Baume (degree)	*DS* % (m/m)	Density (kg/m^3)	Density (lb/ft^3)	Density (lb/ga)	Baume (degree)
22	1091.83	67.97	9.09	12.2	61	1294.64	80.61	10.78	33.0
23	1096.36	68.26	9.13	12.7	62	1300.59	80.98	10.83	33.5
24	1100.92	68.54	9.16	13.3	63	1306.57	81.35	10.88	34.0
25	1105.51	68.82	9.20	13.8	64	1312.60	81.73	10.93	34.5
26	1110.14	69.11	9.24	14.4	65	1318.66	82.11	10.98	35.0
27	1114.80	69.40	9.28	14.9	66	1324.76	82.49	11.03	35.6
28	1119.49	69.70	9.32	15.5	67	1330.90	82.87	11.08	36.1
29	1124.22	69.99	9.36	16.0	68	1337.08	83.25	11.13	36.6
30	1128.98	70.29	9.40	16.6	69	1343.30	83.64	11.18	37.1
31	1133.78	70.59	9.44	17.1	70	1349.56	84.03	11.23	37.6
32	1138.61	70.89	9.48	17.7	71	1355.85	84.42	11.29	38.1
33	1143.47	71.19	9.52	18.2	72	1362.18	84.82	11.34	38.6
34	1148.37	71.50	9.56	18.7	73	1368.58	85.22	11.39	39.1
35	1153.31	71.80	9.60	19.3	74	1374.96	85.61	11.45	39.5
36	1158.28	72.11	9.64	19.8	75	1381.41	86.02	11.50	40.0
37	1163.29	72.42	9.68	20.4	76	1387.90	86.42	11.55	40.5
38	1168.33	72.74	9.72	20.9	77	1394.42	86.83	11.61	41.0
39	1173.41	73.06	9.77	21.4	78	1400.98	87.24	11.66	41.5
40	1178.53	73.37	9.81	22.0	79	1407.58	87.65	11.72	42.0
41	1183.68	73.70	9.85	22.5	80	1414.21	88.06	11.77	42.5
42	1188.87	74.02	9.90	23.0	81	1420.88	88.48	11.83	43.0
43	1194.10	74.34	9.94	23.6	82	1427.59	88.89	11.88	43.4
44	1199.36	74.67	9.98	24.1	83	1434.34	89.31	11.94	43.9
45	1204.67	75.00	10.03	24.6	84	1441.12	89.74	12.00	44.4
46	1210.01	75.33	10.07	25.2	85	1447.94	90.16	12.05	44.9
47	1215.38	75.67	10.12	25.7	86	1454.80	90.59	12.11	45.3
48	1220.80	76.01	10.16	26.2	87	1461.70	91.02	12.17	45.8
49	1226.25	76.35	10.21	26.8	88	1468.62	91.45	12.23	46.3
50	1231.74	76.69	10.25	27.3	89	1475.59	91.89	12.28	46.7
51	1237.27	77.03	10.30	27.8	90	1482.59	92.32	12.34	47.2
52	1242.84	77.38	10.35	28.3	91	1489.63	92.76	12.40	47.7
53	1248.44	77.73	10.39	28.9	92	1496.71	93.20	12.46	48.1
54	1254.08	78.08	10.44	29.4	93	1503.81	93.64	12.52	48.6
55	1259.76	78.44	10.49	29.9	94	1510.96	94.09	12.58	49.0
56	1265.48	78.79	10.53	30.4	95	1518.14	94.54	12.64	49.5
57	1271.23	79.15	10.58	30.9	96	1525.35	94.99	12.70	49.9
58	1277.03	79.51	10.63	31.5	97	1532.60	95.44	12.76	50.4
59	1282.86	79.88	10.68	32.0	98	1539.88	95.89	12.82	50.8
60	1288.73	80.43	10.75	32.5	99	1547.19	96.35	12.88	51.3

Source: U.S. National Bureau of Standard, Circular C440

Table A.2 Sucrose Solubility in Pure Sucrose Solutions

Temperature (°C)	Sucrose Solubility (g/g)	Sucrose Solubility (% m/m)	Temperature (°C)	Sucrose Solubility (g/g)	Sucrose Solubility (% m/m)	Temperature (°C)	Sucrose Solubility (g/g)	Sucrose Solubility (% m/m)
1	1.82	64.53	41	2.37	70.30	81	3.73	78.88
2	1.83	64.62	42	2.39	70.49	82	3.79	79.11
3	1.83	64.71	43	2.41	70.68	83	3.84	79.32
4	1.84	64.80	44	2.43	70.88	84	3.89	79.53
5	1.85	64.90	45	2.46	71.08	85	3.94	79.74
6	1.86	65.00	46	2.48	71.29	86	3.99	79.95
7	1.87	65.10	47	2.51	71.50	87	4.04	80.16
8	1.87	65.21	48	2.53	71.71	88	4.09	80.37
9	1.88	65.32	49	2.56	71.92	89	4.15	80.57
10	1.89	65.43	50	2.59	72.13	90	4.20	80.77
11	1.90	65.55	51	2.61	72.33	91	4.25	80.97
12	1.91	65.67	52	2.64	72.54	92	4.31	81.17
13	1.92	65.79	53	2.67	72.75	93	4.37	81.37
14	1.93	65.91	54	2.70	72.96	94	4.42	81.56
15	1.94	66.04	55	2.73	73.18	95	4.48	81.74
16	1.96	66.18	56	2.76	73.39	96	4.53	81.92
17	1.97	66.31	57	2.79	73.61	97	4.59	82.10
18	1.98	66.45	58	2.82	73.83	98	4.65	82.30
19	1.99	66.59	59	2.85	74.05	99	4.71	82.48
20	2.01	66.74	60	2.89	74.27	100	4.77	82.66
21	2.02	66.86	61	2.92	74.48			
22	2.03	67.01	62	2.95	74.70			
23	2.04	67.16	63	2.99	74.92			
24	2.06	67.31	64	3.02	75.14			
25	2.07	67.47	65	3.06	75.36			
26	2.09	67.63	66	3.10	75.59			
27	2.10	67.79	67	3.14	75.82			
28	2.12	67.95	68	3.17	76.04			
29	2.14	68.13	69	3.21	76.27			
30	2.15	68.30	70	3.25	76.48			
31	2.17	68.47	71	3.29	76.70			
32	2.19	68.64	72	3.33	76.92			
33	2.21	68.81	73	3.37	77.13			
34	2.23	69.00	74	3.42	77.36			
35	2.24	69.18	75	3.46	77.58			
36	2.26	69.35	76	3.51	77.81			
37	2.28	69.54	77	3.55	78.04			
38	2.30	69.73	78	3.60	78.25			
39	2.32	69.92	79	3.64	78.46			
40	2.34	70.10	80	3.69	78.68			

Calculated based on Eq. (3.16.2)

Table A.3 Sucrose Solubility in Impure Sucrose Solutions (g sucrose/g water)

Purity (%)	Temperature (°C)														
	20	25	30	35	40	45	50	55	60	65	70	75	80	85	90
100	2.01	2.07	2.15	2.24	2.34	2.46	2.59	2.73	2.89	3.06	3.21	3.46	3.69	3.94	4.20
99	2.00	2.06	2.14	2.23	2.33	2.44	2.57	2.71	2.87	3.04	3.25	3.43	3.66	3.90	4.16
98	1.99	2.05	2.13	2.22	2.32	2.43	2.56	2.69	2.85	3.02	3.19	3.41	3.63	3.87	4.13
97	1.98	2.04	2.12	2.21	2.31	2.42	2.54	2.68	2.83	3.00	3.17	3.39	3.61	3.85	4.11
96	1.97	2.03	2.11	2.20	2.30	2.41	2.53	2.67	2.82	2.99	3.16	3.38	3.60	3.83	4.09
95	1.96	2.03	2.11	2.19	2.29	2.40	2.52	2.66	2.81	2.98	3.15	3.36	3.59	3.82	4.08
94	1.96	2.02	2.10	2.19	2.28	2.39	2.52	2.65	2.81	2.97	3.14	3.36	3.58	3.82	4.07
93	1.95	2.02	2.10	2.18	2.28	2.39	2.51	2.65	2.80	2.97	3.14	3.35	3.58	3.81	4.07
92	1.95	2.01	2.09	2.18	2.27	2.38	2.51	2.64	2.80	2.96	3.13	3.35	3.57	3.81	4.07
91	1.95	2.01	2.09	2.18	2.27	2.38	2.51	2.64	2.80	2.96	3.13	3.35	3.58	3.82	4.08
90	1.94	2.01	2.09	2.17	2.27	2.38	2.51	2.64	2.80	2.96	3.14	3.36	3.58	3.82	4.09
89	1.94	2.01	2.09	2.17	2.27	2.38	2.51	2.64	2.80	2.97	3.14	3.36	3.59	3.83	4.10
88	1.94	2.01	2.09	2.17	2.27	2.38	2.51	2.65	2.80	2.97	3.14	3.37	3.60	3.85	4.11
87	1.94	2.01	2.09	2.18	2.27	2.38	2.51	2.65	2.81	2.98	3.15	3.38	3.61	3.86	4.13
86	1.94	2.01	2.09	2.18	2.28	2.39	2.52	2.66	2.81	2.98	3.16	3.39	3.62	3.88	4.15
85	1.95	2.01	2.09	2.18	2.28	2.39	2.52	2.66	2.82	2.99	3.17	3.40	3.64	3.90	4.17
84	1.95	2.02	2.09	2.18	2.28	2.40	2.53	2.67	2.83	3.00	3.18	3.42	3.66	3.92	4.20
83	1.95	2.02	2.10	2.19	2.29	2.40	2.53	2.68	2.84	3.01	3.19	3.43	3.67	3.94	4.22
82	1.95	2.02	2.10	2.19	2.29	2.41	2.54	2.68	2.85	3.02	3.21	3.45	3.69	3.96	4.25
81	1.96	2.03	2.11	2.20	2.30	2.41	2.55	2.69	2.86	3.04	3.22	3.46	3.71	3.98	4.28
80	1.96	2.03	2.11	2.20	2.30	2.42	2.56	2.70	2.87	3.05	3.24	3.48	3.74	4.01	4.31
79	1.97	2.04	2.12	2.21	2.31	2.43	2.57	2.71	2.88	3.06	3.25	3.50	3.76	4.03	4.34
78	1.97	2.04	2.12	2.22	2.32	2.44	2.57	2.72	2.89	3.08	3.27	3.52	3.78	4.06	4.37
77	1.98	2.05	2.13	2.22	2.33	2.45	2.58	2.74	2.91	3.09	3.29	3.54	3.80	4.11	4.40
76	1.98	2.05	2.14	2.23	2.34	2.46	2.60	2.75	2.92	3.11	3.30	3.56	3.83	4.18	4.54

Table A.3 Continued

Purity (%)	Temperature (°C)														
	20	25	30	35	40	45	50	55	60	65	70	75	80	85	90
75	1.99	2.06	2.14	2.24	2.34	2.47	2.61	2.76	2.93	3.13	3.34	3.62	3.90	4.26	4.62
74	1.99	2.07	2.15	2.25	2.35	2.48	2.62	2.77	2.95	3.14	3.36	3.65	3.93	4.30	4.69
73	2.00	2.07	2.16	2.25	2.36	2.49	2.63	2.79	2.96	3.16	3.38	3.67	4.02	4.36	4.75
72	2.01	2.08	2.17	2.26	2.37	2.50	2.64	2.80	2.98	3.18	3.47	3.69	4.09	4.46	4.86
71	2.01	2.09	2.17	2.27	2.38	2.51	2.65	2.81	2.99	3.20	3.50	3.77	4.15	4.53	4.94
70	2.02	2.09	2.18	2.28	2.39	2.52	2.67	2.83	3.05	3.30	3.57	3.88			
69	2.03	2.10	2.19	2.29	2.40	2.53	2.68	2.84	3.10	3.32	3.60	3.93			
68	2.04	2.11	2.20	2.30	2.41	2.54	2.69	2.86	3.14	3.34	3.68	4.02			
67	2.04	2.12	2.21	2.31	2.42	2.55	2.71	2.87	3.18	3.36	3.75	4.09			
66	2.05	2.13	2.22	2.32	2.44	2.57	2.78	2.98	3.22	3.44	3.82	4.16			
65	2.06	2.17	2.27	2.38	2.52	2.66	2.83	3.05	3.28	3.56	3.89	4.27			
64	2.07	2.19	2.29	2.42	2.55	2.71	2.88	3.10	3.32	3.64	3.94	4.37			
63	2.08	2.21	2.31	2.44	2.58	2.74	2.92	3.14	3.37	3.72	4.04	4.46			
62	2.14	2.23	2.34	2.47	2.61	2.78	2.97	3.20	3.44	3.77	4.14	4.56			
61	2.16	2.26	2.36	2.49	2.64	2.81	3.02	3.25	3.52	3.85	4.23	4.69			
60	2.19	2.28	2.40	2.53	2.69	2.87	3.08	3.32	3.61	3.95	4.35	4.81			
59	2.20	2.30	2.43	2.57	2.73	2.92	3.13	3.39	3.69	4.04	4.46	4.95			
58	2.23	2.34	2.47	2.61	2.76	2.97	3.20	3.46	3.78	4.15	4.58	5.11			
57	2.26	2.38	2.50	2.65	2.81	3.02	3.26	3.54	3.87	4.26	4.72	5.27			

Calculated based on Eq. (3.16.5)

Table A.4 Boiling Point Elevation (*BPE*) of Pure Sucrose Solutions

DS (% m/m)	Boiling Point of Water (°C)						
	60	70	80	90	100	110	120
10	0.08	0.08	0.09	0.09	0.10	0.11	0.12
15	0.15	0.16	0.18	0.19	0.21	0.22	0.24
20	0.25	0.27	0.29	0.31	0.34	0.35	0.37
25	0.37	0.39	0.42	0.45	0.48	0.51	0.54
30	0.50	0.54	0.56	0.62	0.66	0.71	0.75
35	0.63	0.70	0.75	0.81	0.86	0.92	0.98
40	0.84	0.90	0.96	1.04	1.11	1.20	1.28
45	1.07	1.16	1.24	1.34	0.42	1.52	1.64
50	1.37	1.46	1.57	1.66	1.79	1.91	2.04
55	1.82	1.92	2.04	2.22	2.36	2.50	2.68
60	2.34	2.52	2.70	2.89	3.09	3.31	3.54
62	2.52	2.71	2.90	3.11	3.33	3.56	3.80
64	2.74	2.95	3.16	3.39	3.62	3.88	4.14
66	3.02	3.24	3.48	3.73	3.99	4.27	4.56
68	3.36	3.61	3.87	4.15	4.44	4.75	5.07
70	3.77	4.05	4.34	4.65	4.98	5.33	5.69
72	4.26	4.57	4.91	5.26	5.63	6.02	6.43
74	4.84	5.19	5.57	5.97	6.39	6.83	7.30
76	5.51	5.92	6.35	6.81	7.28	7.79	8.32
78	6.30	6.76	7.26	7.77	8.32	8.90	9.51
80		7.74	8.30	8.89	9.52	10.20	10.90
82			9.5	10.2	10.9	11.6	12.4
84				11.6	12.4	13.3	14.2
86					14.2	15.2	16.2
88						17.3	18.5
90							21.0

Source: From U.S. National Bureau of Standard, Circular C440 and abstracted from *Sugar Technologists Manual* with the permission of Bartens

Table A.5 Boiling Point Elevation (*BPE*) of Impure Sucrose Solutions

Purity (%)	*DS* (% m/m)	Boiling Point of Water (°C)						
		60	70	80	90	100	110	120
90	10	0.09	0.09	0.10	0.11	0.11	0.12	0.13
	20	0.29	0.31	0.33	0.36	0.38	0.41	0.43
	30	0.57	0.61	0.66	0.71	0.76	0.81	0.86
	40	0.97	1.04	1.12	1.20	1.28	1.37	1.47
	50	1.59	1.71	1.83	1.96	2.10	2.25	2.40
	60	2.65	2.85	3.05	3.27	3.50	3.75	4.00
	65	3.24	3.48	3.74	4.00	4.29	4.58	4.90
	70	4.22	4.54	4.87	5.21	5.58	5.97	6.38
	75	5.73	6.16	6.60	7.08	7.57	8.10	8.65
	80		8.52	9.14	9.79	10.5	11.2	12.0
	85				13.6	14.5	15.5	16.6
	90							22.8
80	10	0.10	0.11	0.12	0.12	0.13	0.14	0.15
	20	0.34	0.36	0.39	0.41	0.44	0.47	0.51
	30	0.67	0.72	0.77	0.82	0.88	0.94	1.01
	40	1.13	1.22	1.31	1.40	1.50	1.60	1.71
	50	1.85	1.99	2.14	2.29	2.45	2.62	2.80
	60	3.02	3.24	3.48	3.73	3.99	4.27	4.56
	65	3.69	3.96	4.25	4.56	4.88	5.21	5.57
	70	4.79	5.14	5.52	5.91	6.33	6.77	7.23
	75	6.47	6.95	7.45	7.99	8.55	9.14	9.77
	80	8.91	9.57	10.3	11.0	11.8	12.6	13.5
	85			14.2	15.2	16.3	17.4	18.6
	90						23.8	25.5
70	10	0.12	0.13	0.14	0.15	0.16	0.17	0.18
	20	0.40	0.43	0.46	0.49	0.53	0.56	0.60
	30	0.79	0.84	0.91	0.97	1.04	1.11	1.19
	40	1.33	1.43	1.53	1.64	1.76	1.88	2.01
	50	2.18	2.35	2.52	2.70	2.89	3.09	3.30
	60	3.46	3.71	3.98	4.27	4.57	4.88	5.22
	65	4.22	4.53	4.86	5.21	5.58	5.96	6.37
	70	5.47	5.88	6.31	6.76	7.23	7.73	8.26
	75	7.39	7.94	8.51	9.12	9.76	10.40	11.20
	80	10.2	10.9	11.7	12.6	13.4	14.4	15.4
	85		15.1	17.2	18.5	19.8	21.1	22.6
	90				23.7	25.4	27.2	29.0

Source: From *Sugar Technologists Manual* with the permission of Bartens

Table A.6 Specific Heat Capacity (C_P) of Pure Sucrose Solutions (kJ/kg.°C)

Temperature (°C)	Sucrose Content (%)								
	0	10	20	30	40	50	60	70	80
0	4.19	3.94	3.69	3.43	3.18	2.93	2.68	2.43	2.18
10	4.19	3.94	3.70	3.46	3.21	2.97	2.73	2.48	2.24
20	4.19	3.95	3.72	3.48	3.24	3.01	2.77	2.54	2.30
30	4.19	3.96	3.73	3.50	3.27	3.04	2.82	2.59	2.36
40	4.19	3.97	3.75	3.52	3.30	3.08	2.86	2.64	2.42
50	4.19	3.97	3.76	3.55	3.33	3.12	2.91	2.69	2.48
60	4.19	3.98	3.78	3.57	3.36	3.16	2.95	2.75	2.54
70	4.19	3.99	3.79	3.59	3.39	3.19	3.00	2.80	2.60
80	4.19	4.00	3.81	3.61	3.42	3.23	3.04	2.85	2.66
90	4.19	4.00	3.82	3.64	3.45	3.27	3.09	2.90	2.72
100	4.20	4.01	3.83	3.66	3.48	3.30	3.12	2.95	2.77
110	4.21	4.02	3.85	3.68	3.51	3.33	3.15	3.00	2.83
120	4.22	4.03	3.87	3.70	3.54	3.36	3.18	3.05	2.89
130	4.23	4.04	3.89	3.72	3.57	3.39	3.21	3.10	2.95

Note: Values for **T** > 90°C were obtained by interpolation of the graph
Calculated based on Eq. (1.18)

Table A.7 Specific Heat Capacity (C_P) of Impure Sucrose Solutions (kJ/kg.°C)

Temp. (°C)	Purity (P) = 90% Dry-Substance Content (%)								Purity (P) = 80% Dry-Substance Content (%)							
	10	20	30	40	50	60	70	80	10	20	30	40	50	60	70	80
0	3.93	3.68	3.42	3.16	2.91	2.65	2.40	2.14	4.26	3.67	3.41	3.15	2.89	2.63	2.37	2.11
10	3.94	3.69	3.44	3.19	2.95	2.70	2.45	2.20	3.93	3.68	3.43	3.18	2.92	2.67	2.42	2.17
20	3.95	3.71	3.47	3.22	2.98	2.74	2.50	2.26	3.94	3.70	3.45	3.21	2.96	2.72	2.47	2.23
30	3.95	3.72	3.49	3.25	3.02	2.79	2.56	2.32	3.95	3.71	3.47	3.24	3.00	2.76	2.52	2.29
40	3.96	3.74	3.51	3.28	3.06	2.83	2.61	2.38	3.96	3.73	3.50	3.27	3.04	2.81	2.58	2.35
50	3.97	3.75	3.53	3.31	3.10	2.88	2.66	2.44	3.96	3.74	3.52	3.30	3.07	2.85	2.63	2.41
60	3.98	3.77	3.56	3.34	3.13	2.92	2.71	2.50	3.97	3.76	3.54	3.33	3.11	2.90	2.68	2.47
70	3.98	3.78	3.58	3.37	3.17	2.97	2.77	2.56	3.98	3.77	3.56	3.36	3.15	2.94	2.73	2.53
80	3.99	3.80	3.60	3.40	3.21	3.01	2.82	2.62	3.99	3.79	3.59	3.39	3.19	2.99	2.79	2.59
90	4.00	3.81	3.62	3.43	3.25	3.06	2.87	2.68	3.99	3.80	3.61	3.42	3.22	3.03	2.84	2.65
100	4.01	3.83	3.65	3.46	3.28	3.10	2.92	2.74	4.00	3.82	3.63	3.45	3.26	3.08	2.89	2.71
110	4.01	3.84	3.67	3.49	3.32	3.15	2.98	2.80	4.01	3.83	3.65	3.48	3.30	3.12	2.94	2.77
120	4.02	3.86	3.69	3.52	3.36	3.19	3.03	2.86	4.02	3.85	3.68	3.51	3.34	3.17	3.00	2.83
130	4.03	3.87	3.71	3.55	3.40	3.24	3.08	2.92	4.02	3.86	3.70	3.54	3.37	3.21	3.05	2.89

Table A.7 Continued

Temp. (°C)	Purity (P) = 70% Dry-Substance Content (%)								Purity (P) = 90% Dry-Substance Content (%)							
	10	20	30	40	50	60	70	80	10	20	30	40	50	60	70	80
0	3.92	3.66	3.39	3.13	2.86	2.60	2.33	2.07	3.92	3.65	3.38	3.11	2.84	2.57	2.30	2.03
10	3.93	3.67	3.42	3.16	2.90	2.64	2.39	2.13	3.93	3.66	3.40	3.14	2.88	2.62	2.35	2.09
20	3.94	3.69	3.44	3.19	2.94	2.69	2.44	2.19	3.93	3.68	3.42	3.17	2.92	2.66	2.41	2.15
30	3.94	3.70	3.46	3.22	2.98	2.73	2.49	2.25	3.94	3.69	3.45	3.20	2.95	2.71	2.46	2.21
40	3.95	3.72	3.48	3.25	3.01	2.78	2.54	2.31	3.95	3.71	3.47	3.23	2.99	2.75	2.51	2.27
50	3.96	3.73	3.51	3.28	3.05	2.82	2.60	2.37	3.96	3.72	3.49	3.26	3.03	2.80	2.56	2.33
60	3.97	3.75	3.53	3.31	3.09	2.87	2.65	2.43	3.96	3.74	3.51	3.29	3.07	2.84	2.62	2.39
70	3.97	3.76	3.55	3.34	3.13	2.91	2.70	2.49	3.97	3.75	3.54	3.32	3.10	2.89	2.67	2.45
80	3.98	3.78	3.57	3.37	3.16	2.96	2.75	2.55	3.98	3.77	3.56	3.35	3.14	2.93	2.72	2.51
90	3.99	3.79	3.60	3.40	3.20	3.00	2.81	2.61	3.99	3.78	3.58	3.38	3.18	2.98	2.77	2.57
100	4.00	3.81	3.62	3.43	3.24	3.05	2.86	2.67	3.99	3.80	3.60	3.41	3.22	3.02	2.83	2.63
110	4.00	3.82	3.64	3.46	3.28	3.09	2.91	2.73	4.00	3.81	3.63	3.44	3.25	3.07	2.88	2.69
120	4.01	3.84	3.66	3.49	3.31	3.14	2.96	2.79	4.01	3.83	3.65	3.47	3.29	3.11	2.93	2.75
130	4.02	3.85	3.69	3.52	3.35	3.18	3.02	2.85	4.02	3.84	3.67	3.50	3.33	3.16	2.98	2.81

Calculated based on Eq. (1.18)

Table A.8 Specific Enthalpy (*H*) of Pure Sucrose Solutions (kJ/kg)

Temp. (°C)	Sucrose Content (%)								
	0	10	20	30	40	50	60	70	80
10	41.9	39.4	36.9	34.5	32.0	29.5	27.0	24.6	22.1
20	83.7	78.9	74.0	69.1	64.3	59.4	54.5	49.7	44.8
30	125.6	118.4	111.2	104.0	96.8	89.6	82.5	75.3	68.1
40	167.5	158.0	148.6	139.2	129.7	120.3	110.8	101.4	92.0
50	209.4	197.7	186.1	174.5	162.9	151.3	139.7	128.1	116.5
60	251.2	237.5	223.8	210.1	196.4	182.7	169.0	155.3	141.5
70	293.1	277.4	261.6	245.9	230.2	214.4	198.7	183.0	167.2
80	335.0	317.3	299.6	281.9	264.2	246.6	228.9	211.2	193.5
90	376.8	357.3	337.7	318.2	298.6	279.1	259.5	240.0	220.4
100	418.7	397.4	376.0	354.7	333.3	312.0	290.6	269.3	247.9
110	462	437	414	392	367	345	322	299	275
120	506	477	452	429	403	378	353	329	304
130	549	517	490	466	438	412	386	359	333

Note: Values for **T** > 100°C were obtained by interpolation of the graph
Calculated based on Eq. (1.19)

Table A.9 Specific Enthalpy (H) of Impure Sucrose Solutions (kJ/kg.°C)

Temp. (°C)	Purity (P) = 90% Dry-Substance Content (%)								Purity (P) = 80% Dry-Substance Content (%)							
	10	20	30	40	50	60	70	80	10	20	30	40	50	60	70	80
10	39	37	34	32	29	27	24	22	39	37	34	32	29	26	24	21
20	79	74	69	64	59	54	49	44	79	74	69	64	58	53	48	43
30	118	111	104	96	89	82	74	67	118	111	103	96	88	81	73	66
40	158	148	139	129	119	110	100	90	158	148	138	128	118	109	99	89
50	198	186	174	162	150	138	126	115	197	185	173	161	149	137	125	113
60	237	223	209	195	181	167	153	139	237	223	208	194	180	166	151	137
70	277	261	245	229	213	197	181	165	277	260	244	228	211	195	178	162
80	317	299	281	263	245	227	209	191	317	298	280	261	243	224	206	188
90	357	337	317	297	277	257	237	217	356	336	316	295	275	255	234	214
100	397	375	353	331	310	288	266	244	396	374	352	330	307	285	263	241
110	437	413	390	366	343	319	296	272	436	412	388	364	340	316	292	268
120	477	452	427	401	376	351	326	300	477	451	425	399	373	347	322	296
130	517	491	464	437	410	383	356	329	517	489	462	434	407	379	352	324

Table A.9 Continued

Temp. (°C)	Purity (P) = 70% Dry-Substance Content (%)								Purity (P) = 90% Dry-Substance Content (%)							
	10	20	30	40	50	60	70	80	10	20	30	40	50	60	70	80
10	39	37	34	31	29	26	24	21	39	37	34	31	29	26	23	21
20	79	73	68	63	58	53	48	43	79	73	68	63	58	52	47	42
30	118	110	103	95	88	80	72	65	118	110	102	95	87	79	71	64
40	157	147	138	128	118	108	98	88	157	147	137	127	117	106	96	86
50	197	185	172	160	148	136	123	111	197	184	172	159	147	134	122	109
60	237	222	208	193	179	164	149	135	236	222	207	192	177	162	148	133
70	276	260	243	226	210	193	176	160	276	259	242	225	208	191	174	157
80	316	297	279	260	241	222	203	185	316	297	278	258	239	220	201	182
90	356	335	314	294	273	252	231	210	356	334	313	292	271	250	228	207
100	396	373	351	328	305	282	260	237	396	372	349	326	303	280	256	233
110	436	411	387	362	338	313	288	264	435	410	385	360	335	310	285	260
120	476	450	423	397	371	344	318	291	476	449	422	395	368	341	314	287
130	516	488	460	432	404	376	348	320	516	487	458	430	401	372	344	315

Calculated based on Eq. (1.19)

Table A.10 Properties of Saturated Steam and Water (in Metric and English systems)

Temperature (°C)	Vapor pressure (kPa)	Enthalpy			Temperature (°F)	Vapor pressure (lb/in.^{2})	Enthalpy		
		Saturated Vapor (H_V) (kJ/kg)	Liquid (H_C) (kJ/kg)	Vaporization (λ) (kJ/kg)			Saturated Vapor (H_V) (Btu/lb)	Liquid (H_C) (Btu/lb)	Vaporization (λ) (Btu/lb)
0	0.6	2501.6	0.0	2501.6	32	0.1	1075.4	0.0	1075.4
5	0.9	2510.7	21.0	2489.7	35	0.1	1076.7	3.0	1073.7
10	1.2	2519.9	42.0	2477.9	40	0.1	1078.9	8.0	1070.9
15	1.7	2528.9	62.9	2466.0	45	0.2	1081.1	13.0	1068.1
20	2.2	2538.2	83.9	2454.3	50	0.2	1083.3	18.1	1065.2
25	3.2	2547.3	104.8	2442.5	55	0.2	1085.5	23.1	1062.4
30	4.3	2556.3	125.8	2430.5	60	0.3	1087.7	28.1	1059.6
35	5.6	2565.4	146.6	2418.8	65	0.3	1089.9	33.1	1056.8
40	7.4	2574.3	167.6	2406.7	70	0.4	1092.0	38.1	1053.9
45	9.6	2583.2	188.5	2394.7	75	0.4	1094.2	43.1	1051.1
50	12.4	2592.1	209.3	2382.8	80	0.5	1096.4	48.1	1048.3
55	15.8	2600.9	230.2	2370.7	85	0.6	1098.6	53.1	1045.5
60	19.9	2609.6	251.1	2358.5	90	0.7	1100.7	58.1	1042.6
65	25.0	2618.3	272.1	2346.2	95	0.8	1102.9	63.1	1039.8
70	31.2	2626.8	293.0	2333.8	100	1.0	1105.0	68.1	1037.0
75	38.6	2635.3	313.9	2321.4	110	1.3	1109.3	78.0	1031.3
80	47.4	2643.7	334.9	2308.8	120	1.7	1113.5	88.0	1025.5
85	57.8	2651.9	355.9	2296.0	130	2.2	1117.8	98.0	1019.8
90	70.1	2660.1	376.9	2283.2	140	2.9	1121.9	108.0	1013.9
95	84.6	2668.1	398.0	2270.1	150	3.7	1126.1	118.0	1008.1
100	101.4	2676.1	419.0	2257.1	160	4.8	1130.1	128.0	1002.1

Table A.10 Continued

Temperature (°C)	Vapor pressure (kPa)	Enthalpy: Saturated Vapor (H_V) (kJ/kg)	Enthalpy: Liquid (H_C) (kJ/kg)	Enthalpy: Vaporization (λ) (kJ/kg)	Temperature (°F)	Vapor pressure (lb/in.^{2})	Enthalpy: Saturated Vapor (H_V) (Btu/lb)	Enthalpy: Liquid (H_C) (Btu/lb)	Enthalpy: Vaporization (λ) (Btu/lb)
105	120.8	2683.8	440.2	2243.7	170	6.0	1134.2	138.0	996.2
110	143.3	2691.5	461.3	2230.2	180	7.5	1138.2	148.0	990.2
115	169.1	2699.0	482.5	2216.5	190	9.3	1142.1	158.0	984.1
120	198.5	2706.3	503.7	2202.6	200	11.5	1145.9	168.1	977.8
125	232.1	2713.5	525.0	2188.5	210	14.1	1149.7	178.1	971.6
130	270.1	2720.5	546.3	2174.2	220	17.2	1153.5	188.2	965.3
135	313.0	2727.3	567.7	2159.6	230	20.8	1157.1	198.3	958.8
140	316.3	2733.9	589.1	2144.8	240	25.0	1160.7	208.4	952.3
145	415.4	2740.3	610.6	2129.7	250	29.8	1164.2	218.6	945.6
150	475.8	2746.5	632.2	2114.3	260	35.4	1167.6	228.8	938.8
155	543.1	2752.4	653.8	2098.6	270	41.9	1170.9	239.0	932.0
160	617.8	2758.1	675.6	2082.6	280	49.2	1174.1	249.2	924.9
165	700.5	2763.5	697.3	2066.2	290	57.5	1177.2	259.4	917.8
170	791.7	2768.7	719.2	2049.5	300	67.0	1180.2	269.7	910.5
175	892.0	2773.6	741.2	2032.4	310	77.6	1183.0	280.1	902.9
180	1002	2778.2	763.2	2015.0	320	89.6	1185.8	290.4	895.4
190	1254	2786.4	807.6	1978.8	340	117.9	1190.8	311.3	879.5
200	1554	2793.2	852.5	1940.8	350	134.5	1193.1	321.8	871.3
225	2548	2803.3	966.8	1836.5	360	152.9	1195.2	332.4	862.9
250	3973	2801.5	1085.4	1716.1	370	173.2	1197.2	343.0	854.2
275	5942	2785.0	1210.1	1574.9	380	195.6	1199.0	353.6	845.4
300	8581	2749.0	1344.0	1405.0	390	220.2	1200.6	364.3	836.3

Source: Abstracted from *Steam Tables* by Keenan et al. (1969), with the permission of John Wiley and Sons, Inc.

Table A.11 Density (kg/m³) of Massecuites at Different Crystal Contents and Temperatures

DS Massecuite (% m/m)	*DS* Mother Liquor (% m/m)	Crystal Content (%)	Temperature (°C)					
			40	50	60	70	80	90
88	75	52.0	1472.4	1468.4	1464.3			
	76	50.0	1471.9	1467.8	1463.6	1459.2		
	77	47.8	1471.2	1467.1	1462.8	1458.4		
	78	45.5	1470.5	1466.3	1461.9	1457.4	1452.8	
	79	42.9	1469.7	1465.4	1460.9	1456.3	1451.6	
	80	40.0	1468.8	1464.4	1459.8	1455.1	1450.3	1445.5
89	76	54.2	1480.6	1476.7	1472.6	1468.5		
	77	52.2	1480.0	1476.0	1471.9	1467.7		
	78	50.0	1479.3	1475.3	1471.1	1466.8	1462.4	
	79	47.6	1478.6	1474.4	1470.1	1465.8	1461.3	
	80	45.0	1477.7	1473.5	1469.1	1464.6	1460.1	1455.5
	81	42.1	1476.8	1472.5	1468.0	1463.4	1458.7	1454.1
90	77	56.5	1488.8	1485.0	1481.1	1477.1		
	78	54.5	1488.2	1484.4	1480.4	1476.3	1472.1	
	79	52.4	1487.5	1483.6	1479.5	1475.3	1471.1	
	80	50.0	1486.8	1482.7	1478.6	1474.3	1470.0	1465.7
	81	47.4	1485.9	1481.8	1477.5	1473.2	1468.7	1464.3
	82	44.4	1485.0	1480.7	1476.4	1471.9	1467.3	1462.8
91	78	59.1	1497.3	1493.6	1489.8	1485.9	1482.0	
	79	57.1	1496.6	1492.9	1489.0	1485.1	1481.0	
	80	55.0	1495.9	1492.1	1488.2	1484.1	1480.0	1475.9
	81	52.6	1495.2	1491.2	1487.2	1483.1	1478.9	1474.7
	82	50.0	1494.3	1490.3	1486.1	1481.9	1477.6	1473.3
	83	47.1	1493.3	1489.2	1484.9	1480.6	1476.2	1471.8
92	79	61.9	1505.8	1502.3	1498.6	1494.9	1491.1	
	80	60.0	1505.2	1501.6	1497.9	1494.1	1490.2	1486.3
	81	57.9	1504.5	1500.8	1497.0	1493.1	1489.2	1485.2
	82	55.6	1503.7	1499.9	1496.0	1492.1	1488.0	1484.0
	83	52.9	1502.8	1498.9	1494.9	1490.9	1486.7	1482.6
	84	50.0	1501.8	1498.7	1493.7	1489.5	1485.3	1481.0
93	81	63.2	1514.0	1510.5	1506.9	1503.3	1499.6	1495.9
	82	61.1	1513.3	1509.7	1506.1	1502.3	1498.6	1494.8
	83	58.8	1512.5	1508.8	1505.1	1501.3	1497.4	1493.6
	84	56.3	1511.6	1507.8	1504.0	1500.1	1496.1	1492.2
	85	53.3	1510.5	1506.7	1502.7	1498.7	1494.6	1490.6
	86	50.0		1505.4	1501.3	1497.1	1492.9	1488.8
94	84	62.5	1521.5	1518.0	1514.4	1510.8	1507.2	1503.5
	85	60.0	1520.6	1517.0	1513.4	1509.6	1505.9	1502.1
	86	57.1		1515.9	1512.1	1508.3	1504.4	1500.5
	87	53.8			1510.6	1506.7	1502.7	1498.7
	88	50.0			1508.9	1504.8	1500.6	1496.5
	89	45.5				1502.6	1498.2	1494.0

Source: From *Sugar Technologists Manual* with the permission of Bartens

Table A.12 Viscosity (in cp) of Pure Sucrose Solutions

DS (% m/m)	Temperature (°C)								
	0	5	10	15	20	25	30	35	40
20	3.8	3.2	2.7	2.3	2.0	1.7	1.5	1.3	1.2
21	4.0	3.3	2.8	2.4	2.0	1.8	1.6	1.4	1.2
22	4.2	3.5	2.9	2.5	2.1	1.9	1.6	1.4	1.3
23	4.4	3.7	3.1	2.6	2.2	1.9	1.7	1.5	1.3
24	4.7	3.9	3.2	2.7	2.3	2.0	1.8	1.6	1.4
25	5.0	4.1	3.4	2.9	2.5	2.1	1.9	1.6	1.5
26	5.3	4.3	3.6	3.0	2.6	2.2	2.0	1.7	1.5
27	5.6	4.6	3.8	3.2	2.7	2.4	2.1	1.8	1.6
28	5.9	4.8	4.0	3.4	2.9	2.5	2.2	1.9	1.7
29	6.3	5.1	4.3	3.6	3.0	2.6	2.3	2.0	1.8
30	6.7	5.5	4.5	3.8	3.2	2.8	2.4	2.1	1.8
31	7.2	5.8	4.8	4.0	3.3	2.9	2.5	2.2	1.9
32	7.7	6.2	5.1	4.3	3.6	3.1	2.7	2.3	2.0
33	8.3	6.7	5.5	4.6	3.8	3.3	2.8	2.5	2.2
34	8.9	7.2	5.8	4.9	4.1	3.5	3.0	2.6	2.3
35	9.6	7.7	6.3	5.2	4.4	3.7	3.2	2.7	2.4
36	10.4	8.3	6.7	5.6	4.7	3.9	3.4	2.9	2.5
37	11.3	9.0	7.3	6.0	5.0	4.2	3.6	3.1	2.7
38	12.3	9.7	7.8	6.4	5.4	4.5	3.8	3.3	2.9
39	13.4	10.6	8.5	6.9	5.8	4.8	4.1	3.5	3.0
40	14.7	11.5	9.2	7.5	6.2	5.2	4.4	3.8	3.3
41	16.1	12.6	10.1	8.2	6.7	5.6	4.7	4.0	3.5
42	17.8	13.9	11.0	8.9	7.3	6.1	5.1	4.3	3.7
43	19.7	15.3	12.1	9.7	7.9	6.6	5.5	4.7	4.0
44	21.9	16.9	13.3	10.6	8.6	7.1	6.0	5.0	4.3
45	24.5	18.7	14.7	11.7	9.4	7.8	6.4	5.4	4.6
46	27.4	20.9	16.2	12.9	10.4	8.5	7.0	5.9	5.0
47	30.8	23.3	18.0	14.2	11.4	9.2	7.7	6.4	5.4
48	34.8	26.2	20.1	15.8	12.6	10.2	8.4	7.0	5.9
49	39.5	29.5	22.6	17.6	14.0	11.3	9.2	7.7	6.4
50	45.1	33.4	25.4	19.7	15.5	12.5	10.2	8.4	7.0
51	51.6	38.0	28.7	22.1	17.4	13.9	11.3	9.2	7.7
52	59.5	43.5	32.6	24.9	19.5	15.5	12.5	10.2	8.5
53	69.0	50.0	37.2	28.3	22.0	17.4	13.9	11.4	9.4
54	80.5	57.8	42.7	32.2	24.9	19.5	15.6	12.7	10.4
55	94.5	67.3	49.3	36.9	28.3	22.1	17.6	14.2	11.6
56	111.8	78.8	57.2	42.5	32.4	25.1	19.8	15.9	13.0
57	133.2	93.0	66.9	49.3	37.2	28.7	22.5	18.0	14.5
58	160.1	110.0	78.7	57.6	43.1	33.0	25.7	20.4	16.4
59	193.9	132.0	93.4	67.6	50.2	38.1	29.5	23.2	18.6
60	237.4	160.0	111.7	80.1	58.9	44.5	34.1	26.7	21.2
61	293.7	195.6	134.7	95.6	69.7	52.0	39.6	30.8	24.3
62	367.2	241.2	164.1	115.1	83.0	61.4	46.4	35.7	28.0
63	464.9	300.7	201.8	139.9	99.8	73.0	54.7	41.8	32.5
64	595.7	379.2	250.8	171.6	121.0	87.6	65.0	49.2	38.0

Table A.12 Continued

DS (% m/m)	Temperature (°C)								
	0	5	10	15	20	25	30	35	40
65	773.1	484.1	315.3	212.8	148.2	106.1	77.9	58.4	44.7
66	**1020**	**626**	**401**	**267**	183.3	129.7	94.1	69.9	53.0
67	**1365**	**822**	**517**	**339**	229.4	160.2	114.9	84.2	63.4
68	**1859**	**1096**	**677**	**436**	290.5	200.1	141.7	103.0	76.5
69	**2579**	**1486**	**899**	**568**	372.7	252.9	176.8	126.9	93.2
70	**3654**	**2052**	**1214**	**752**	485.0	323.9	223.2	158.1	114.8

Source: U.S. National Bureau of Standard, Circular C440
Note: Boldface values are extrapolated

Table A.13 Viscosity (in Pa.s) of Massecuites at Different Crystal Contents (*CC*) and Temperatures

DS Massecuite (% m/m)	*DS* Mother Liquor (%)	*CC* Massecuite (%)	Temperature (°C)					
			40	50	60	70	80	90
Purity = 90%								
90	76	58.3	199	97	52	30		
	77	56.5	155	73	38	22	13	
	78	54.6		58	29	16	9.5	
	79	52.4		48	23	12	7.1	4.4
91	82	50.0				73	38	21
	83	47.1				53	26	14
	84	43.8					20	10
92	83	52.9				53	26	14
	84	50.0					20	10
	85	46.7						7.6
93	83	58.8				300	151	82
	84	56.3					91	48
	85	53.3						30
Purity = 80%								
90	78	54.6	116	54	28	16		
	79	52.6	99	44	22	12	7.0	
	80	50.0	89	38	18	10	5.4	2.6
91	80	55.0	296	122	59	31	18	
	81	52.6		99	46	23	13	7.5
	82	50.0		84	37	18	9.7	5.5
92	82	55.6		318	140	69	37	21
	83	52.9			105	49	25	14
93	84	56.3				177	87	46
	85	53.3				120	57	29

Table A.13 Continued

DS Massecuite (% m/m)	*DS* Mother Liquor (%)	*CC* Massecuite (%)	Temperature (°C)					
			40	50	60	70	80	90
Purity = 70%								
90	80	50.0	80	35	17	9.2		
	81	47.4	74	31	15	7.6		
	82	44.4	73	29	13	6.6	3.6	
91	81	52.6	214	90	43	22		
	82	50.0	190	76	34	17	9.3	
	83	47.1	178	68	29	14	7.4	
92	83	52.9	587	224	97	47	24	
	84	50.0		186	77	35	18	
93	85	53.3		652	254	112	54	28
	86	50.0			196	82	38	19
94	88	50.0				219	90	41
Purity = 60%								
90	82	44.4	64	27	12			
	83	41.2	66	26	11			
	84	37.5	71	26	11	5.2		
91	84	43.8	153	57	24	11		
	85	40.0	162	57	23	10		
	86	35.7	182	60	23	9.7		
92	84	50.0	445	165	70	33		
	85	46.7	418	146	59	27		
93	86	50.0		461	175	75		
	87	46.2		408	145	59	27	
94	88	50.0			423	150	60	27

Source: Abstracted from *Sugar Technologists Manual* with the permission of Bartens

Table A.14 Refractive Index (*RI*) of Pure Sucrose Solutions (at 20°C)

DS (% m/m)	*RI*	*DS* (% m/m)	*RI*	*DS* (% m/m)	*RI*	*DS* (% m/m)	*RI*	*DS* (% m/m)	*RI*
0.0	1.3330	1.6	1.3353	3.2	1.3376	4.8	1.3400	6.4	1.3424
0.2	1.3333	1.8	1.3356	3.4	1.3379	5.0	1.3403	6.6	1.3427
0.4	1.3336	2.0	1.3359	3.6	1.3382	5.2	1.3406	6.8	1.3430
0.6	1.3339	2.2	1.3362	3.8	1.3385	5.4	1.3409	7.0	1.3433
0.8	1.3341	2.4	1.3365	4.0	1.3388	5.6	1.3412	7.2	1.3436
1.0	1.3344	2.6	1.3368	4.2	1.3391	5.8	1.3415	7.4	1.3439
1.2	1.3347	2.8	1.3370	4.4	1.3394	6.0	1.3418	7.6	1.3442
1.4	1.3350	3.0	1.3373	4.6	1.3397	6.2	1.3421	7.8	1.3445

Table A.14 Continued

DS (% m/m)	*RI*	*DS* (% m/m)	*RI*	*DS* (% m/m)	*RI*	*DS* (% m/m)	*RI*	*DS* (% m/m)	*RI*
8.0	1.3448	16.6	1.3583	23.2	1.3692	31.8	1.3843	40.4	1.4005
8.2	1.3451	16.8	1.3586	23.4	1.3696	32.0	1.3847	40.6	1.4008
8.4	1.3454	17.0	1.3589	23.6	1.3699	32.2	1.3851	40.8	1.4012
8.6	1.3457	17.2	1.3592	23.8	1.3703	32.4	1.3854	41.0	1.4016
8.8	1.3460	17.4	1.3596	24.0	1.3706	32.6	1.3858	41.2	1.4020
9.0	1.3463	17.6	1.3599	24.2	1.3709	32.8	1.3861	41.4	1.4024
9.2	1.3466	17.8	1.3602	24.4	1.3713	33.0	1.3865	41.6	1.4028
9.4	1.3469	18.0	1.3605	24.6	1.3716	33.2	1.3869	41.8	1.4032
9.6	1.3472	18.2	1.3609	24.8	1.3720	33.4	1.3872	42.0	1.4036
9.8	1.3475	18.4	1.3612	25.0	1.3723	33.6	1.3876	42.2	1.4040
10.0	1.3478	18.6	1.3615	25.2	1.3726	33.8	1.3879	42.4	1.4044
10.2	1.3481	18.8	1.3619	25.4	1.3730	34.0	1.3883	42.6	1.4048
10.4	1.3485	19.0	1.3622	25.6	1.3733	34.2	1.3887	42.8	1.4052
10.6	1.3488	19.2	1.3625	25.8	1.3737	34.4	1.3891	43.0	1.4056
10.8	1.3491	19.4	1.3628	26.0	1.3740	34.6	1.3894	43.2	1.4060
11.0	1.3494	19.6	1.3632	26.2	1.3744	34.8	1.3898	43.4	1.4064
11.2	1.3497	19.8	1.3635	26.4	1.3747	35.0	1.3902	43.6	1.4068
11.4	1.3500	20.0	1.3638	26.6	1.3751	35.2	1.3906	43.8	1.4072
11.6	1.3503	20.2	1.3642	26.8	1.3754	35.4	1.3909	44.0	1.4076
11.8	1.3506	20.4	1.3645	27.0	1.3758	35.6	1.3913	44.2	1.4080
12.0	1.3509	20.6	1.3648	27.2	1.3760	35.8	1.3916	44.4	1.4084
12.2	1.3512	20.8	1.3652	27.4	1.3765	36.0	1.3920	44.6	1.4086
12.4	1.3516	21.0	1.3655	27.6	1.3768	36.2	1.3924	44.8	1.4092
12.6	1.3519	21.2	1.3659	27.8	1.3772	36.4	1.3928	45.0	1.4096
12.8	1.3522	21.4	1.3662	28.0	1.3775	36.6	1.3931	45.2	1.4100
13.0	1.3525	21.6	1.3665	28.2	1.3779	36.8	1.3935	45.4	1.4104
13.2	1.3528	21.8	1.3669	28.4	1.3782	37.0	1.3939	45.6	1.4109
13.4	1.3531	20.0	1.3638	28.6	1.3786	37.2	1.3943	45.8	1.4113
13.6	1.3535	20.2	1.3642	28.8	1.3789	37.4	1.3947	46.0	1.4117
13.8	1.3538	20.4	1.3645	29.0	1.3793	37.6	1.3950	46.2	1.4121
14.0	1.3541	20.6	1.3648	29.2	1.3797	37.8	1.3954	46.4	1.4125
14.2	1.3544	20.8	1.3652	29.4	1.3800	38.0	1.3958	46.6	1.4129
14.4	1.3547	21.0	1.3655	29.6	1.3804	38.2	1.3962	46.8	1.4133
14.6	1.3550	21.2	1.3659	29.8	1.3808	38.4	1.3966	47.0	1.4137
14.8	1.3554	21.4	1.3662	30.0	1.3811	38.6	1.3970	47.2	1.4141
15.0	1.3557	21.6	1.3665	30.2	1.3815	38.8	1.3974	47.4	1.4145
15.2	1.3560	21.8	1.3669	30.4	1.3818	39.0	1.3978	47.6	1.4150
15.4	1.3563	22.0	1.3672	30.6	1.3822	39.2	1.3982	47.8	1.4154
15.6	1.3566	22.2	1.3675	30.8	1.3825	39.4	1.3986	48.0	1.4158
15.8	1.3570	22.4	1.3679	31.0	1.3829	39.6	1.3989		
16.0	1.3573	22.6	1.3682	31.2	1.3833	39.8	1.3993		
16.2	1.3576	22.8	1.3685	31.4	1.3836	40.0	1.3997		
16.4	1.3579	23.0	1.3689	31.6	1.3840	40.2	1.4001		

Source: U.S. National Bureau of Standard, Circular C440

Table A.15 Bulk Density of Materials Used in Sugar Production

	kg/m^3	lb/ft^3
Beet seed	700	44
Beets, washed	660	41
Beet tare:		
Soil and sand, wet	2100	131
Soil and sand, dry	1600	100
Stones	1400	87
Trash	300	19
Limestone	1500	94
Coke	400	25
Carbonation gas	1.5	0.1
Coal	750	47

Note: Values are approximate

Table A.16 Bulk Density of Products and By-products of a Beet-Sugar Factory

	kg/m^3	lb/ft^3	lb/ga
Granulated-refined sugar	900	56	
Powdered sugar	450	28	
Brown sugar	850	53	
Molasses, 80% *DS*	1400	87	12
Betaine, 55% *DS*	1300	81	11
Raffinate, 60% *DS*	1250	78	10
Pulp:			
Pressed	620	39	
Dry pulp, 90% *DS*	220	14	
Dry molasses, 90% *DS*	200	12	
Pellets, 90% *DS*	650	41	
Carbonation lime residue, 70% *DS*	1100	69	

Note: Values are approximate

Table A.17 Conversion Factors

Acceleration
$1\,ft/s^2 = 0.3048\,m/s^2$
Acceleration of gravity (gravitational force)
$= 32.19\,ft/s^2 = 9.81\,m/s^2$

Area
$1\,ft^2 = 0.2832\,m^2$
1 acre $= 4047\,m^2 = 43515\,ft^2 = 0.405$ hectare
1 hectare $= 10^4 m^2 = 2.471$ acre
$1\,in.^2 = 6.452 \times 10^{-4}\,m^2 = 645.16\,mm^2$
$1\,yd^2 = 9\,ft^2 = 0.837\,m^2$

Density
$1\,lb/ft^3 = 16.019\,kg/m^3$
$1\,lb/ga = 1.198 \times 10^2\,kg/m^3$
$1\,kg/m^3 = 0.062\,lb/ft^3 = 0.0083\,lb/ga$

Diffusivity
$1\,ft^2/hr = 2.581 \times 10^{-5}\,m^2/s$

Energy
1 Btu $= 1.055$ kJ $= 252.16$ cal $= 0.252$ kcal
1 kcal $= 4.184$ kJ
$1\,J = 1\,N.m = 1\,kg.m^2/s^2$
$1\,kWh = 3.6 \times 10^3$ kJ

Enthalpy
1 Btu/lb $= 2.326$ kJ/kg

Force(f)
$1\,lbf = 4.448\,N$
$1\,N = 1\,kg.m/s^2$
$1\,dyne = 1\,g.cm/s^2 = 10^{-5}\,kg.m/s^2$

Heat flow
1 Btu/h $= 0.293$ W
1 Btu/min $= 17.58$ W
$1\,kJ/h = 2.778 \times 10^{-4}\,kW$
$1\,J/s = 1\,W$

Heat flux
$1\,Btu/h.ft^2 = 3.155\,W/m^2$

Heat transfer coefficient
$1\,Btu/h.ft^2.°F = 5.678\,W/m^2.°C = 5.678\,kJ/m^2.°C$
$1\,Btu/h.ft^2.°F = 1.357 \times 10^{-4}\,cal/s.cm^2.°C$

Length
1 ft $= 0.3048$ m
$1\,in. = 2.54 \times 10^{-2}\,m = 2.54\,cm = 25.4\,mm$
1 micron $= 1\,\mu m = 10^{-6}\,m$
$1\,nm = 10^{-9}\,m$
$1\,Å = 1\,\mu m = 10^{-10}\,m$
1 mile $= 1.6093 \times 10^3$ m

Mass
1 lb $= 0.45359$ kg $= 16$ oz
1 oz $= 28.35$ g
1 metric ton $= 10^3$ kg $= 0.9078$ short ton
$1\,g = 10^3\,mg = 61 \times 10^{22}$ Dalton

Power
1 hp $= 745.71$ W $= 550$ ft.lb/s $= 0.707$ Btu/s
1 W $= 1$ J/s $= 14.34$ cal/min
1 kWh $= 3412$ BTU
1 Btu/h $= 0.293$ W

Pressure
1 psi $=$ psig $+ 14.7$
$1\,psi = 6.895\,kPa = 0.069\,bar = 6895 \times 10^3\,N/m^2$
$1\,bar = 10^5\,Pa = 10^5\,N/m$
$1\,Pa = 1\,N/m^2$
1 atm $= 1.0133$ bar $= 14.7$ psi
$1\,atm = 1.0133 \times 10^5\,Pa = 101.33\,kPa = 760\,atm$
$1\,lb/ft^2 = 4.788 \times 10^2\,dyne/cm^2 = 47.88\,N/m^2$

Specific heat
$1\,Btu/lb.°F = 4186\,J/kg.°C = 10^3\,cal/kg.°C$

Temperature
$T_F = 1.8 \times T_C + 32$
$T_C = (T_F - 32)/1.8$
$K = °C = 273$

Thermal conductivity
$1\,Btu/h.ft.°F = 1.731\,W/m.K$
$1\,Btu.in./ft.h.°F = 1.442 \times 10^{-2}\,W/m^2.K$

Velocity
$1\,ft/s^2 = 0.3048\,m/s^2$

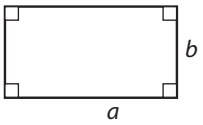
Viscosity
$1\,cp = 10^{-2}\,g/cm.s = 10^{-3}\,kg/m.s$
$1\,cp = 10^{-2}$ poise $= 10^{-3}\,Pa.s = 10^{-3}\,N.s/m^2$
$1\,lb/ft.h = 0.413\,cp$
$1\,lb/ft.s = 1488.16\,cp$
$1\,N.s/m^2 = 1\,Pa.s = 1\,kg/m.s$

Volume
$1\,ft^3 = 7.481$ U.S. ga
1 U.S. ga $= 4$ qt $= 0.083$ British ga
1 U.S. ga $= 3.785\,L = 3.785 \times 10^{-3}\,m^3$
$1\,ft^3 = 0.028\,m^3 = 28.32\,L = 18.28\,in.^2$
1 qt $= 0.946$ L
1 oz $= 29.55$ ml
$1\,m^3 = 10^3\,L = 35.31\,ft^3$
$1\,L = 10^3\,cm^3 = 10^3$ ml

Work
1 hp.h $= 0.746$ kWh $= 2.6845$ J $= 2544.5$ Btu
1 ft.lb $= 1.356$ J

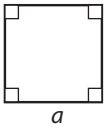
Table A.18 Useful Formulas from Geometry

Rectangle



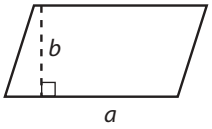
A: area
$A = ab$
Perimeter = sum of all sides

Square



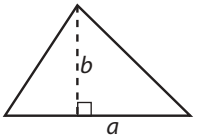
$A = a^2$

Parallelogram



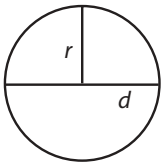
$A = ab$

Triangle



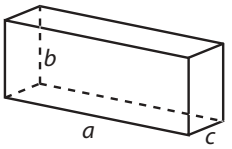
$A = \frac{ab}{2}$

Circle



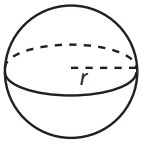
$\pi = 3.14$
r: radius
d: diameter
C: circumfrence
$A = \pi r^2$
$d = 2r$
$C = \pi d$

Rectangular solid



V: volume
S: surface area
$V = abc$
$S = 2\,(ab + ac + bc)$

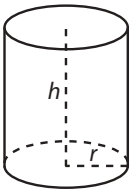
Sphere



$V = \frac{4\pi r^3}{3}$

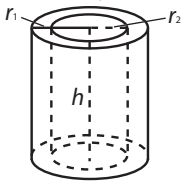
$V = \frac{\pi d^3}{6}$

Cylinder



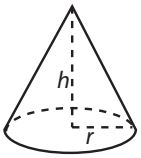
$V = \pi r^2 h$

Hollow cylinder



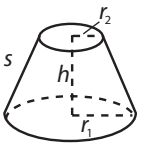
$V = \pi h\,(r_1^2 - r_2^2)$

Cone



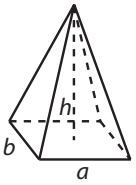
$V = \frac{\pi r^2 h}{3}$

Truncated cone



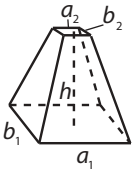
$V = \frac{\pi h}{3}\,(r_1^2 + r_2^2 + r_1 r_2)$

Pyramid



$V = \frac{abh}{3}$

Truncated pyramid



$V = \frac{h\,(a_1 b_1 + a_2 b_2 + \sqrt{(a_1 b_1)(a_2 b_2)})}{3}$

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GLOSSARY

Absolute humidity: The concentration of water vapor in the air
Absolute pressure: Absolute pressure = atmospheric pressure – vacuum pressure
Absolute zero: The lowest possible temperature (–274°C = –460°F = 0 Kelvin)
Absorption: To penetrate something, as when a sponge absorbs water
Acid: A compound that produces hydrogen ions (H) when it is dissolved in water
Acid rain: Generic term for atmospheric precipitation with a pH less than 5
Adant (slab) **sugar**: Molded sugar with a rough surface produced by casting and chopping
Adenosine triphosphate (ATP): The main energy-carrying molecule
Adhesive force: An attractive force between molecules of two polar substances
Adsorption: To stick, or adhere, to the surface of a substance, such as nonsugars (nonsucroses) that stick to the surface of calcium carbonate during juice purification
Aerobic: In the presence of oxygen
Affination: The process of mixing raw sugar or low-purity massecuite with high-purity syrup and then centrifuging it to improve the raw-sugar quality
Agglomeration: The process of gathering two substances together to form a ball-shaped substance
Alcohol: An organic compound that has a hydroxyl group (OH) bounded to an alkane-like carbon atom ROH
Aldehyde: An organic compound containing an aldehyde group (CHO)
Alkalinity: Carbonate (CO_3), bicarbonate (HCO_3), and hydroxide (OH^-) of Ca, Mg, Na, and K
Amino acid: A compound that contains an amino group (NH_2) and a carboxyl group (COOH), both groups joined to the same carbon
Amino group: A functional group containing NH_2 group
Amorphous: A compound containing broken, unorganized, and shapeless molecules
Anaerobic: In the absence of oxygen
Anion: An element with negative-charged ion
Antioxidant: A compound that interact with free radicals and block oxidation process by giving the extra electron, thus preventing it from being taken from a healthy cell (main antioxidants are vitamin A, C, D, E, and K)

Beet-Sugar Handbook, by Mosen Asadi
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Aqueous solution: A solution in which water is the solvent
Ash: Soluble and insoluble salts of organic and inorganic compounds
Atmospheric pressure: The pressure with which the atmosphere presses down on any object; at atmospheric pressure, water boils at 100°C (212°F)
Atom: The smallest particle of an element
Atomic mass (weight)**:** The weighted average mass of an element's atoms
Atomic mass unit (AMU): A convenient unit for describing the mass of an atom (1 AMU = 1/12 the mass of a carbon-12 atom)
Atomic number: The number of protons in an atom
Avogadro's number: The number of units in a mole (6.02×10^{23})

Backboiling: Mixing two syrups of different purity to make feed for a given boiling stage to produce massecuite with the desired purity
Bagasse: Sugarcane fibrous material after being separated from the juice in the mill (or diffuser)
Base (alkali)**:** A compound that produces hydroxide ion (OH) when dissolved in water
Beet brei: A chopped beet-sample prepared by grinding the beets in a food processor for analytical purposes (mainly for sucrose-content determination)
Beet campaign (beet-processing season): The period during which the factory processes beets
Beet-end: The section of the factory comprising the stations from beet precleaning through juice evaporation
Beet fluming: Transporting beets by the force of water
Beet intercampaign: The period between two beet campaigns
Beet laboratory (tare laboratory)**:** A laboratory for determination of tare and for testing sugarbeets to determine growers' payment for delivered beets
Beet pulp: See *Pulp*
Beet tare: Clay, sand, stone, and trash mixed with the beets
Betaine: One of the components of sugarbeet that ends up in molasses during sugarbeet processing
Biennial plant: A plant requiring two years for a full life cycle
Bioethanol: Ethanol produced from a plant source
Biomass: Raw material from plants and animals
Blowdown of boiler: The process of opening the boiler's blowdown valve for a short period to remove suspended materials
BOD (biochemical oxygen demand): The amount of oxygen, expressed in mg/L (ppm), required to biologically oxidize the organic substances in a sample over time (usually five days)
Boiling point (*BP*): The temperature at which boiling starts at atmospheric pressure
Boiling-point elevation (*BPE*): Increase of a solution's boiling point above that of pure water
Brix: See *RDS*
Brown sugar (soft sugar): A mixture of fine granulated-refined sugar and brown cane sugar syrup, with a pleasant cane-molasses flavor
Buffer: A weak acid and its salt or a weak base and its salt–if present in a given solution, the buffer can keep pH almost constant when small amounts of acid or base are added to the solution or when the solution goes under evaporation
Bulk density: Mass per volume of loose materials

Cake: The solid retained on filter during the filtration of a solution containing large precipitate
Cake filters: Filter presses or rotary-vacuum filters that are used to further concentrate carbonation mud to produce cake, which is known as carbonation-lime residue (limecake)
Calendria: The heating section of an evaporator consisting of heating tubes
Calorie: A metric unit of heat energy (the amount of energy required to raise the temperature of 1 g of water 1°C)
Campaign: Beet-processing time in beet factories
Capacity (of a beet-sugar factory): A factory's capability to process sugarbeet in a 24-hour period
Caramel: The product of a caramelization reaction
Caramelization: A process in which sucrose decomposes to glucose, fructose, and finally to caramel at temperatures close to its melting point (approximately 185°C)
Carbohydrates: Sugars, starches, and dietary fibers composed of carbon, hydrogen, and oxygen
Carbonation: Adding carbonation gas (gas rich in CO_2) to the juice
Carbonation gas: The gas produced in the limekiln containing about 33% CO_2
Carbonation-lime residue (limecake): The used lime produced from further concentration of carbonation mud in cake filters (filter presses or rotary drum filters) and one of the by-products of the beet-sugar factory–it is thicker than carbonation slurry
Carbonation mud: The solids separated from the first-carbonation slurry produced after gasing in the clarifiers or thickening filters
Carbonation process: The process of mixing carbonation gas with juice during the juice-purification process
Carbonation slurry: A slurry consisting of juice and carbonation precipitate (i.e., first-carbonation slurry or second-carbonation slurry)
Carbonyl group: A functional group containing a CO or CHO group
Carboxyl group: A functional group containing a COOH group
Carboxylic acids: Acids containing a COOH group, such as methanoic acid (HCOOH, known as formic acid)
Carcinogenic: A substance capable of causing cancer
Catalyst: A substance that speeds up a reaction without being consumed
Cation: An element with a positive-charged ion
Caustic: A common name for sodium hydroxide, $Ca(OH)_2$
Centrifugal force (F_C): The force produced by high-speed rotation of a centrifuge basket
Centrifuge: A machine that separates solids from liquid by centrifugal force
Charles's law: The volume (V) occupied by a sample of a gas is directly proportional to its temperature (T) at constant pressure, $V_1/T_1 = V_2/T_2$
Chelate: A molecule that can form more than one covalent bond with a metal ion
Chemical change: A change that affects the chemical property of a substance to create new (in a chemical change, new substances are formed that have different properties)
Chemical formula (molecular formula): Gives the symbol and number of the atoms of each compound
Chlorination (of water): The use of hypochlorous acid (HOCl) to disinfect water
Chlorophyll: The green pigment molecules in plants responsible for photosynthesis
Chromatography: A separation technique based on separating solutes between a stationary and mobile phase
Chromosome: Part of the cell that contains genes

Clarifier: Equipment used in the settling process
CMS (concentrated molasses solids)**:** See *Raffinate*
Coagulation: The process of neutralizing and destabilizing colloids
COD (chemical oxygen demand): The rough amount of oxygen equal to the amount of organic substances in wastewater
Coefficient of variation (*CV*): A way of describing the **precision** of the result of a set of measurements (a low *CV* value characterizes a better uniformity, and a high *CV* value indicates a wide and poor distribution size); *CV* of less than 35% is desirable for sugar crystals
Cogeneration of heat and power (CHP): The combination of steam and power production to use in sugarbeet processing
Colloids: High molecular substances such as pectin, dextran, coloring materials, decaying beet particles, and microorganisms in sugarbeet processing
Combustion reaction: The chemical reaction of combustible components of fuel with oxygen from air
Condensate: Pure water obtained by condensation of steam or vapor
Condenser: Equipment for the condensation of steam or vapor, using water as a cooling medium
Condenser water: Mixture of condensate and cooling water produced by a condenser
Cone sugar (loaf sugar): a cone-shaped, molded sugar with a smooth surface produced by a pressing or casting process using a special mold
Conversion factor: An expression of the relationship between two units (see the Appendix)
Cooling crystallization: Lowering the temperature of the massecuite causing the water in the mother liquor to hold less sugar
Cossettes: Beet slices produced by a beet slicer
Covalent bond: A bond formed by sharing electrons between atoms
Cross-links: Covalent bonds linking the chains in a polymer, usually by divinylbenzen (DVB)
Crown: A part of the beet to which the petioles are attached
Crystal: A solid with plane faces at definite angles and with atoms or molecules in a regular three-dimensional arrangement
Crystal lattice: The three-dimensional, ordered, repeating pattern of molecules in a crystal
Crystallization: The production of crystals from a solution, leaving impurities behind
Crystallization effect: The difference in purity between the feed to the pan and the mother liquor centrifuged from the massecuite produced in the pan
Crystallization rate: The mass of a substance crystallized in 1 minute on $1\,m^2$ of the crystal surface
Crystallizer: Equipment for the crystallization process
Cube sugar: A cubic shapes of casting sugar produced by pressing moist granulated-refined (GR) sugar in a mold

Defeco: Abbreviation for defecation (purification)
Defeco carbonation: Simultaneous addition of lime and carbonation gas to the juice
Dehydration reaction (condensation reaction): A reaction in which one molecule of water is eliminated
Denaturation (as used in sugar technology): Application of heat to change the nature of a sugarbeet cells' protoplasm by coagulation of its main component (protein) to make it permeable
Density: The mass of a substance per its unit volume

Detergent: A synthetic material made from petroleum that washes away dirt—is a surface-active material (surfactant) because it acts to reduce the surface tension of water
Dextran: A long-chain, gelatinous, water-soluble polysaccharide made up of many glucose units
Dextrose equivalent: The relative amount of glucose, fructose, and other reducing sugars in blended products
Diabetes: A disease in which the body either does not produce insulin or does not use it properly to allow glucose (blood sugar) to be converted to energy; sugar instead builds up in the blood creating high blood sugar
Diatomaceous earth: A natural form of silica used as a filter aid in filtration
Diffuser: A large, agitated tank in which cossettes slowly move from one end to the other and the hot water moves in the opposite direction to diffuse the juice from the cossettes
Diffusion efficiency: Degree of desugaring of the cossettes during the diffusion process
Diffusion juice: The product of the diffusion station and feed to the purification station
Diffusion process (as used in beet-sugar industry): The process of moving diffusing components from a higher concentrated phase (inside the beet cells) to a lower concentrated phase (outside the beet cells) through the cell wall—in the diffusion process, the concentration difference of solutes is the driving force
Disaccharate: A salt composed of two oxides with sucrose, for example, $C_{12}H_{22}O_{11}.2CaO$
Disaccharide: A carbohydrate composed of two monosaccharides
Draft (diffusion draft): The mass of diffusion juice produced per mass of beets processed, expressed as percent on beet
Dry pulp: The pulp after it is dried in pulp dryers, containing about 10% moisture
Dry substance (*DS*): The solid content of a solution expressed as mass percentage (% m/m)

Emulsifier: A substance that is soluble in two substances that are insoluble in each other, and it enables them to form an emulsion
Emulsion: A colloid in which both phases are liquid
Endothermic reaction: A reaction that needs energy
Energy: The capacity to do work or supply heat
Enthalpy (*H*, also known as total heat content): The total heat content (the sum of sensible and latent heat) of a substance at constant pressure
Entrainment: The carrying of material droplets by vapor into the condenser during evaporation
Enzyme: A protein or other molecule that acts as a biological catalyst
Evaporation: The process of concentrating a solution by boiling to convert part of its volatile liquid to vapor
Exhaust steam: Steam released from the exhaust of a turbine
Exothermic reaction: A reaction that releases energy (heat) to its surroundings
Extract: The product of the molasses desugaring process with high purity (90+%)
Extract molasses: Molasses produced from extract processing
Extraction process (as defined in the unit operations of chemical engineering): The process of separating one or more solutes by the use of a solvent that *selectively* dissolves one of the solutes—in the extraction process, the solubility difference of solutes is the driving force

Fermentable carbohydrates: Saccharides (sugars) that promote the growth of microorganisms in the fermentation process, such as glucose and fructose

Fermentation process: The sugar-destroying actions of microbes that produce alcohol
Fiber (nondigestible carbohydrates or dietary fiber): Dietary fibers consisting of soluble and insoluble fibers, such as cellulose and hemicellulose
Filter aid: A material used to improve the filtration process
Filter medium: Porous material, such as filter cloth, filter paper, or a metal screen, used to filter solutions
Filter press: A filter that uses pressure to residue filter and press the mud to produce cake and desweeten the cake to produce carbonation-lime with a dry-substance content of about 70%
Filter residue: The solid remaining on the filter medium during filtration
Filtrate: The liquid passed through a filter medium and collected after filtration
Filtration: The process of separating solid particles from a liquid by the force of the pressure difference on both sides of the filter medium
Filtration rate: The measure of the volume of liquid to be filtered per surface area of a filter medium per unit time
First-order reaction: A reaction in which the reaction rate is proportional to the concentration of reactant
Flashing evaporation: A type of evaporation in which vaporization is achieved by heating and pressure reduction
Flavonoids: Dietary compounds including flavones, flavonols, and anthocyanins that have antioxidant properties, and are found in tea, vegetables, fruits, and red wine
Floc (acid beverage floc, ABF in the beverage industry): The cloudy and turbid situation that forms in some sugar-sweetened carbonated soft drinks after standing for several days
Flocculation: The process of massing (agglomeration) coagulated particles, forming larger particles that can be easily separated by sedimentation or filtration
Flume water: Water used to transport beets in a beet flume
Foam: A gas dispersed in a liquid or solid–a gas dispersed in a solid is called "solid foam"
Free radicals: Atoms that miss an electron and are highly reactive, and can be formed when oxygen interacts with certain molecules
Functional group: A group of atoms in a molecule that behaves as a unit and is responsible for that molecule's reactions with other substances, for example, hydroxyl (OH), carbonyl (CO), carboxyl (COOH), or sulfonic (SO_3H)

Gauge pressure: The measurement of pressure relative to atmospheric pressure
Gene: A unit of heredity in the chromosome, which consists of deoxyribonucleic acid (DNA)
Generator: A machine that changes mechanical energy into electricity
Genetic engineering: A new technique (based on adding desirable traits to plants or animals) capable of producing new varieties of plants or animals more quickly than conventional breeding
Genetically modified (GM) **sugarbeets**: Sugarbeets produced based on a genetic-engineering technique
Global warming: Temperature increase on the Earth in recent decades
Glycemic index: The index that compares how much a given sugar increases a person's blood glucose level relative to the increase when pure glucose is ingested, with glucose increase as a standard value of 100
Glycogen (known as **animal starch**): A glucose polymer stored in tissue to be used later
Glycosidic bond: An oxygen bond from one monosaccharide unit to another

Granulated-refined (GR) **sugar**: Crystalline and refined sugar of market quality (table sugar)
Gravitational force (F_G or force of Earth's gravity)**:** Is 9.81 m/s^2 (32 ft/s^2), meaning that an object falling near the Earth's surface increases in speed by 9.81 m/s in each second (s)
Greenhouse gases: Air pollutants that accumulate in the atmosphere, affecting global warming

Hard molasses: Molasses with a hardness content more than 3.0 *mEq*/100 *DS* (or 85 mg/100 *DS*)
Hardness (limesalts): Soluble salts of calcium (Ca) and magnesium (Mg)
Heat: The flow of energy that flows from a hotter object to a colder one because of a temperature difference
Heat exchangers (heaters): Equipment for transferring heat from a high-temperature liquid to a lower-temperature liquid, and vice versa
Heat of combustion: See *Heat value*
Heat-transfer coefficient (*U*): The amount of heat that passes a unit of surface (1 m^2 in metric or 1 ft^2 in English units) in one hour to create a temperature difference of one degree
Heat value (*HV*): The quantity of heat released by combustion of a mass unit of a fuel
Heterogeneous mixture: A mixture composed of two or more substances, all of which retain their identity after they are mixed and can be seen as individual substances
Homogeneous mixture: A mixture composed of two or more substances, none of which retains its identity after they are mixed
Hydrated salts (hydrates): Salts containing one or more molecules of water in their crystalline structure–hydrates formulas are written with a *dot* between the water molecule and the substance to emphasize that intact water molecules are present (e.g., $CaSO_4.2H_2O$ known as gypsum)
Hydrocarbon: An organic compound composed of only carbon and hydrogen
Hydrolysis: The reaction of a compound with water
Hydrometer: A device that works based on the density of the solution sample to measure the percentage of dry substance (*DS*) by mass percentage
Hydroscopic substance: A substance having the ability to absorb water from the surroundings
Hydroxyl group: A functional group containing a OH group
Hypoglycemia: Lower-than-normal glucose in the blood

Ideal gas: A gas that obeys the gas laws under all conditions based on the equation $PV = nRT$
Ideal solution: A solution that obeys Rault's law (an expression of changes in partial pressure of a solution due to dry substance) at different temperatures (the ideal condition is more often found in diluted solutions)
In-process products: A term used in this book to denote the juices, syrups, liquors**,** and masse-cuites as the products of individual stations in a beet-sugar factory
Intermolecular force (I_F): The force that holds molecules together due to chemical bonding
Inversion rate: The measure of the change in concentration per unit time
Invert sugar: The 50 : 50 solution of glucose (dextrose) and fructose (levulose) produced from sucrose inversion
Ion: An atom or a molecule that has lost or gained an electron and become an electrical charge
Ion exchange resin: Synthetic resin composed of organic polymers
Ionic bond: A bond formed by losing or gaining electrons between ions of opposite charge
Ionic compound: A compound that decomposes to positive and negative ions when dissolved in water, such as salt (NaCl)
Isomers: Compounds with the same composition (molecular formula) but different structures

Jam sugar: A mixture of granulated-refined sugar, citric acid (as an acidifying agent), and fruit pectin (as gelling agent) in different ratio—jam sugar is used to make jam
Joule: A unit of heat energy
Juice: An impure sugar solution with a dry-substance content up to 55%
Juice campaign: The period during which stored products (thick juice, standard liquor, or extract [product of molasses-desugaring process]) are processed

Ketone: An organic compound containing a ketone group (CO)
Kinetic energy (E_K): The energy due to the motion of an object
Knife block (box): A metal box used to hold several knives in the drum of a slicer

Latent heat: The heat that changes the temperature and state of a substance
Law of conservation of energy: Mater can be neither created nor destroyed in any chemical or physical change
Lime pond: The area for storage carbonation-lime residue (limecake) produced during juice purification
Limesalts: See *Hardness*
Liming: Adding milk of lime [$Ca(OH)_2$] to the diffusion juice
Liquid: A substance that has a definite volume but changes shape to fill its container
Liquid blend sugars: Liquids that contain other carbohydrates in addition to sugar and invert sugar
Liquid invert sugar: A solution of sugar and invert sugar in water with a definite concentration
Liquid sugar: A solution of sugar in water or sugar and invert sugar in water with a definite concentration
Liquor: A juice concentrated to a definite dry-substance content (by melting some raw sugar in it), filtered, sometimes even decolorized, and used as feed in the crystallization station
Live steam: High-pressure steam from a boiler
Loaf sugar: See *Cone sugar*

Magma: A mixture of sugar crystals and syrup in the affination process
Maillard reaction (Browning reaction): The reaction that occurs between the hydroxyl group of reducing sugars and the amino groups of amino acids at high temperatures to produce melanoidine, which creates high color
Marc (beet marc): Insoluble substances (e.g., pectin and cellulose) of the sugarbeet root
Mass: A measure of the quantity (amount) of matter
Mass number: The number of protons and neutrons in an atom
Massecuite: A semi-solid mixture of crystals and mother liquor
Mean aperture (MA): The size of the screen opening through which 50% (one-half) of the sample crystals fall
Melanization reaction: The reaction of amino acids by enzymes, such as polyphenol oxidase present in beet juice
Melanoidine: Coloring material formed by the Maillard reaction
Melassigenic: A substance (nonsugars) that increases the amount of molasses
Melassigenic coefficient (K_M): The sucrose to nonsucrose ratio in molasses (each kg nonsugars caries about 1.5 kg sugar to molasses)

Melting point: The temperature at which a substance changes from a solid to a liquid
Meniscus: The curving of the water surface at the interface between it and its container
Metastable zone: The region of supersaturation in which crystals already present grow, but new crystal formation (nucleation) does not readily occur
Methyl group: The CH_3 group
Milk-of-lime (MOL or simply lime): A suspension of $Ca(OH)_2$ produced by mixing of quicklime with water in the slaker of the sugar factory
Mingling: A term used by refiners to indicate the process of mixing raw sugar with syrup
Mixture: A heterogeneous (nonuniform) mixture of two or more substances that retain their identity after they are mixed
Molality (*m*, molal concentration): The concentration of a solution expressed as the number of moles of solute in 1 kg of solution
Molar mass: The mass of one mole (each mole of an element consists of 6.02×10^{23} atoms) of an element in grams (numerically, molar mass equals to the molecular mass of the element)
Molarity (*M*, molar concentration): The concentration of a solution expressed as the number of moles of a substance (solute) in 1 liter (L) of solution (solvent)
Molasses: The mother liquor (runoff syrup) from the final stage of crystallization with considerable amounts of sugar left in it
Molasses exhaustion: Lowering molasses to the lowest possible purity
Mole: Expresses the quantity (number) of the mass of a substance in grams (numerically, a mole is the mass of 6.02×10^{23} atoms, molecules, or ions)
Molecular formula: The chemical formula that gives the number of atoms of each compound
Molecular mass (weight): The sum of the atomic masses of all atoms in a molecule
Molecule: A neutral matter consisting of two or more atoms, joined together chemically
Monomer: A small molecule that is used to prepare a polymer
Monosaccharate: A salt composed of an oxide with sucrose, for example, $C_{12}H_{22}O_{11}.CaO$
Monosaccharide: The simplest carbohydrate because it cannot be further decomposed to a simpler carbohydrate
Mother liquor: The liquor from which crystals are formed and that stays with the crystals until it is separated by centrifuging

Neutralization reaction: The reaction of an acid with a base
Neutron: An electrically neutral particle of an atom
Nitration: The substitution of a nitro group (NO_2)
Nitrification bacteria: The bacteria that convert ammonia to nitrate in aerobic systems
Nomenclature: The system of names and formulas used to identify all chemicals
Nonionic compound: A compound that does not decompose to ions when dissolved in water (e.g., sucrose)
Nonmetal: An element that is a poor conductor of heat and electricity
Nonreducing sugars: Sugars (saccharides) in which the carbonyl groups are blocked, so they cannot reduce oxidizing agents
Nonremovable nonsucroses (NRNS): The nonsucroses (nonsugars) that are not removed during purification process
Nonseparable nonsucroses (NSNS): The components of molasses that are difficult to separate from sucrose in the molasses desugaring by chromatographic process

Nonsucrose substances (nonsugars): All soluble substances of beet juice except sucrose (sugar), known as "impurities" among sugar technologists
Normal molasses: A molasses with about 82% dry substance and the lowest purity determined experimentally in the lab for a given factory
Normality (*N*): The concentration of a solution expressed as the number of equivalent mass (*Eq*) of substance (solute) in 1 L of solution
Nucleic acid: A polymeric compound carried by every cell and involved in the transformation of genetic heritage

Organic chemistry: The study of carbon-containing compounds
Organic compound: A compound that contains carbon
Osmosis process: The selective passage (diffusion) of a solvent through a semipermeable membrane from a lower concentration side to a higher concentration side
Oxidation: The loss of an electron (or electrons) by an element or a compound
Oxidation-reduction reaction: A reaction in which electrons are transferred from one atom to another
Oxidizing agent: A substance (e.g., the Fehling reagent) that accepts electron and therefore can oxidize another substance and becomes reduced

Partial liming: The liming process in which less lime is used (compared with complete liming process), so part of invert sugar is not destroyed, and ends up in raw sugar
Particulate matters (PM)**:** Tiny solid particles suspended in air
Parts per billion (ppb): Parts in a billion parts; 1 ppb = 1 micrograms per liter
Parts per millions (ppm): The number of parts in a million parts; 1 ppm = 1 milligrams per liter
Pelleted pulp (pellet): The dry pulp after pelleting in a pulp pelletizer
Percent by mass (weight): The mass of solute divided by the total mass of solution times 100
Percent by volume: The volume of solute divided by the total volume of solution times 100
Permeable: A material that has extremely small pores that allows the passage of small molecules
Petiole: A leafstalk (the footstalk of a leaf, by which it is attached to the stem)
pH: The measure of the acidity or basicity of a solution and is expressed on a scale of 0 to 14
Photosynthetic process: A complex reaction in plants by which carbon dioxide (CO_2) and water (H_2O) react together to produce saccharides (sugars) by absorbing energy from sunlight
Physical change: A change that does not affect the chemical property of a substance (changing water into steam is a physical change from one state of matter into another)
Physical property: A property that can be determined without changing the chemical property of a substance
Pol (abbreviation for **polarization**): A term used in sugar technology with the same meaning as % polarimetric sucrose
Polarimetric sucrose: The sucrose content measured by polarimeter expressed as mass percentage
Polymer: A large molecules formed by linking many small molecules
Polysaccharides: Carbohydrates composed of more than 10 monosaccharides
Potential energy: The energy that occurs because of the position, composition (bonding), shape, or chemical composition of an object

Powdered sugar: A mixture of ground granulated-refined sugar and an anticaking agent (usually starch added to prevent lumping)
Precipitate: A solid that forms in a solution during a chemical reaction
Precoating: A layer of solid, such as a filter aid or calcium carbonate, to help filtration
Press water: The juice squeezed from the wet pulp in a pulp press and returned to the diffuser
Pressed pulp: The pulp after pressing (it contains about 70% moisture)
Pressure: The force acting on a unit of area
Proof test: Putting a small amount of syrup between two fingers and then moving the fingers apart until the syrup breaks
Pulp: Sugarbeet fibrous and sugar-depleted material after being separated from the juice in the diffuser
Purity: A sugar term used to describe the percentage (by mass) of sucrose in the total dry substance (*DS*) of an in-process product
Purity drop (crystallization effect): Difference between purity of feed to the pan and mother liquor centrifuged from the massecuite

Raffinate (CMS for concentrated molasses solids)**:** A byproduct of the molasses desugaring by chromatographic process with a high nonsugar content
Rate of ionization of resin functional group: Dissociation of the functional group of resin into ions (cations or anions) when the resin placed in a solution
Raw sugar: Nonrefined sugar consisting of crystals covered with a thin layer of low-purity syrup (in a beet-sugar factory, the crystalline sugars produced in the second and third stages of the crystallization process are called raw sugar)
RDS (refractometric dry substance, also called Brix): The dry-substance content measured by refractometer expressed as mass percentage
Reducing agent: A substance that gives electrons and therefore can reduce another substance and becomes oxidized
Reducing sugars: Sugars (saccharides) in which the carbonyl groups are free, so they can reduce mild oxidizing agents such as cupric ions (Cu^{2+}) in the Fehling reagent to cuprous ions (Cu^{+})
Reduction: The gain of an electron (or electrons) by an element or a compound
Refinery molasses: Molasses produced from raw cane-sugar processing
Refining raw sugar: Purification of raw sugar through recrystallization and other processes
Refractometer: An instrument for measuring the refractive index and relating it to the dry-substance content of the sample
Regeneration: Washing resin with a NaOH or H_2SO_4 solution to prepare it for further use
Relative humidity: The amount of water vapor in the air (at a given temperature) divided by the amount of water vapor required for saturation of that air, expressed as a percentage
Remelt: The redissolved raw sugar for the crystallization process
Remelt liquor (syrup): Liquor produced from raw sugars melted in a syrup
Removable nonsucroses (RNS): The nonsucroses (nonsugars) removed during purification
Rendement: A term used by raw-sugar refiners to indicate yield (extraction), which is the percentage (by mass) of the refined sugar produced per raw sugar processed
Resin capacity (resin exchange capacity): The ability of resin to remove ions
RNA (ribonucleic acid): The form of nucleic acid present in all parts of the cell, involved in protein synthesis

Rotary drum filter: A filter that uses vacuum to filter and vacuum the mud and desweeten the cake to produce carbonation-lime residue with a dry-substance content of about 50%

Saccharides (sugars): Members of carbohydrates including mono-, di-, tri-, and polysaccharides
Salt: An ionic compound formed from reaction of an acid with a base
Satisfactory molasses: A well-exhausted molasses with the lowest practical purity (60% or less) that can be produced in a given factory with adequate methods and equipment
Saturated solution: A solution containing the maximum amount of dissolved solute possible
Saturated steam (dry saturated steam): Steam without water at the boiling point (when all water is converted into steam)
Saturation coefficient (K_S): Indicates how many times sucrose is more soluble in an impure sucrose solution compared with a pure sucrose solution at the same temperature
Secondary (extract) **molasses**: Molasses produced from processing extract to sugar
Sedimentation: The rough separation of solid particles from a liquid by gravity
Sedimentation rate: The measure of height of the settled particles per unit time
Seed magma: A mixture of crystals and mother liquor used for seeding
Seeding: The introduction of seed to a supersaturated solution in the crystallization process to function as a base for crystal growth
Semipermeable: A material that allows the passage of small particles but not larger ones
Sensible heat: The heat that changes the temperature of a substance but not its state
Significant figures: The number of meaningful digits used to express a value
Silin number: The length (in meter) of 100 g of cossettes
Slab sugar: See *Adant sugar*
Slacking: The mixing of quicklime with water or a diluted juice (often sweet water from filters) in the slaker to produce milk of lime (MOL)
Sludge: The settled mud from the clarifier
Soft molasses: Molasses containing hardness equal, or below, 3 *mEq*/100 *DS* (or 85 mg /100 *DS*, which equates to 850 ppm)
Softener: Softening columns with pipes, and auxiliary equipment (e.g., pumps, tanks, and instrumentation devices)
Softening column: Vertical drum 3 to 5 m in height, 2 to 3 m in diameter, filled with resin
Solid: A substance that has a definite shape and volume
Solubility: The maximum amount of a solute that dissolves in a certain amount of water at certain temperature (g sugar per g water)
Solubility coefficient: The maximum amount of sucrose dissolved in 1 g of water at a given temperature
Solute: The dissolved substance of a solution
Solution: A homogeneous (uniform) mixture composed of two or more substances, none of which retains its identity after they are mixed
Solvent: The liquid in which solutes are dissolved
Special crystal-size sugar: Granulated-refined sugar with small or large crystals that are used in the production of some sugar-added products
Specialty sugars: All sugar products except normal crystal-size granulated-refined (GR) sugar
Specific density (gravity)**:** The mass of a substance divided by the mass of an equal volume of water at a specified temperature

Specific-heat capacity (*c*, often called simply **specific heat**): The amount of heat needed to raise the temperature of 1 g of a substance by 1°C, without a change in state
Specific heat of condensation: The amount of heat energy released when a gas condenses (this is the opposite of the heat of evaporation)
Specific heat of evaporation: The amount of heat energy needed to completely vaporize a liquid after it has reached its boiling point
Specks: Eye-distinguishable foreign materials in sugar
Standard acid: An acid with known concentration used to determine the concentration of a base
Standard base: A base with known concentration used to determine the concentration of an acid
Standard liquor: The feed to the first crystallization stage (the product of the melter consisting of high-raw and low-raw sugar melted in thick juice)
Standard solution: A solution with a known and exact concentration
Standby power: Power bought from local power plants for factory's needs during the beet inter-campaign, when boilers are shut down, and power is needed for equipment maintenance and sugar warehouse activities
Steam: Water in its vapor state at a high pressure and temperature
Steam turbine: A machine for converting the heat energy in steam to kinetic energy to perform work, such as driving a generator for producing electricity
Steffen process: A type of molasses-desugaring process
Stoichiometric quantity: When all chemicals consumed and produced in a chemical reaction are balanced
Stoichiometry (reads **sto-keo-metry**): The quantitative relations between the reactants and products in a chemical reaction
Strike: Product of a batch crystallization process
Strong acid: An acid that completely ionizes and gives H^+ easily when it is dissolved in water
Strong base: A base that completely dissociates and gives OH^- when it is dissolved in water
Sublimation: Changing directly from a solid state to a gas state without passing through the intermediate liquid state
Sucrase: The enzyme involved in the hydrolysis of sucrose to glucose and fructose
Sucrose: Chemical name for sugar with formula of $C_{12}H_{22}O_{11}$
Sucrose normal solution: 26.00 g sucrose diluted in water to a final volume of 100 ml
Sugar alcohols: The products of reduction of some carbohydrates by reducing agents (e.g., H_2)
Sugar-end: The section of the factory consisting of the crystallization station through sugar drying and the silo
Sugar extraction (sugar yield): A percentage ratio to express the efficiency of a sugar factory based on sugar-on-sugar ratio (sugar produced on sugar entered the factory in beets)
Sugar losses: Unrecovered sucrose mainly left in molasses, pulp, and carbonation–lime residue
Sugar recovery: A percentage ratio to express the efficiency of a sugar factory based on the purity of the thin juice and molasses
Sugar refinery: A facility for processing raw-cane sugar to granulated-refined sugar
Sulfitation: The process of adding sulfur dioxide (SO_2) to the juice to reduce color and prevent color formation in the next steps of the operations
Sulfonic group: The SO_3H group
Superheated (supersaturated) **steam**: Steam that acts as a pure steam (no water left in the steam after all traces of water have been evaporated)

Supersaturated solution: A solution that contains more solute than normally can be dissolved in a saturated solution (supersaturated solution is not in equilibrium and solute crystallizes from it on contact)
Supersaturation coefficient: Indicates how many times sucrose is more soluble in an impure sucrose solution compared with a saturated solution at the same temperature
Surface tension: The force of attraction (in N/m)that causes the surface of a liquid to form a drop, caused by the difference between intermolecular forces existing in molecules at the surface of the liquid and those in the interior, so surface molecules are less stable
Surfactant: A surface-active substance that reduces the surface tension of water
Suspension: A mixture containing solid particles that can settle down
Sweetwater: The liquid discharged from filters during desweetening (washing the filter media with water or steam)
Syrup: The mother liquor of the massecuite that is separated by the centrifuging process and nearly saturated with sugar

Tare: See *Beet tare*
Tare laboratory: See *Beet laboratory*
Temperature: The degree of heat or the measurement of hotness or coldness of an object
Thermostable: A stable substance when exposed to heat
Thick juice: The product of the evaporation station that is fed to the crystallization station
Thickening agent: A substance that becomes a gel when dissolved in a small amount of water
Thin juice: The product of the purification station that is fed to the evaporation station
Titration: The determination of the concentration of a solution by adding a standard solution
Total heat content (enthalpy): See *Enthalpy*
Trisaccharate: A salt composed of three oxides with sucrose, for example, $C_{12}H_{22}O_{11}.3CaO$
Trisaccharide: A carbohydrate that can be hydrolyzed to three monosaccharides
True purity: The purity when the sucrose content is determined by the inversion method (true sucrose) and the dry substance is determined by the refractometric method

Unaccounted losses: The difference between the sugar (sucrose) entering the factory with the beets and the sugar produced and accounted losses (sugar left in pulp, in carbonation-lime residue, and in molasses)
Undersaturated solution: A solution in which the solubility of solute (e.g., sucrose) has not reached its maximum yet

Valance electrons: The electrons in the outer (valence) shell of an atom; they participate in chemical bonding
Vapor: The gas molecules in equilibrium with a liquid
Viscosity: The measure of the resistance of a liquid to flow
Volatile Organic Compounds (VOC)**:** Organic compounds that evaporate easily

Wavelength: The distance between peaks of consecutive waves
Weak acid: An acid that ionizes slightly (less than 5%) and gives H^+ with difficulty
Weak base: A base that reacts with water to produce hydroxide ions to only a slight extent (less than 5%)
Weight: The gravitational force applied to an object

Weighted average: Average giving points to the priorities
Wet pulp: The pulp coming from a diffuser, containing about 90% moisture
White crystallization: The first stage of crystallization during which sugar of market quality is produced
Work: The product of force (F) times distance (L), $W = F \times L$

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*Refers to terms defined in Glossary.
MDC process stands for "molasses desugaring by chromatographic process."

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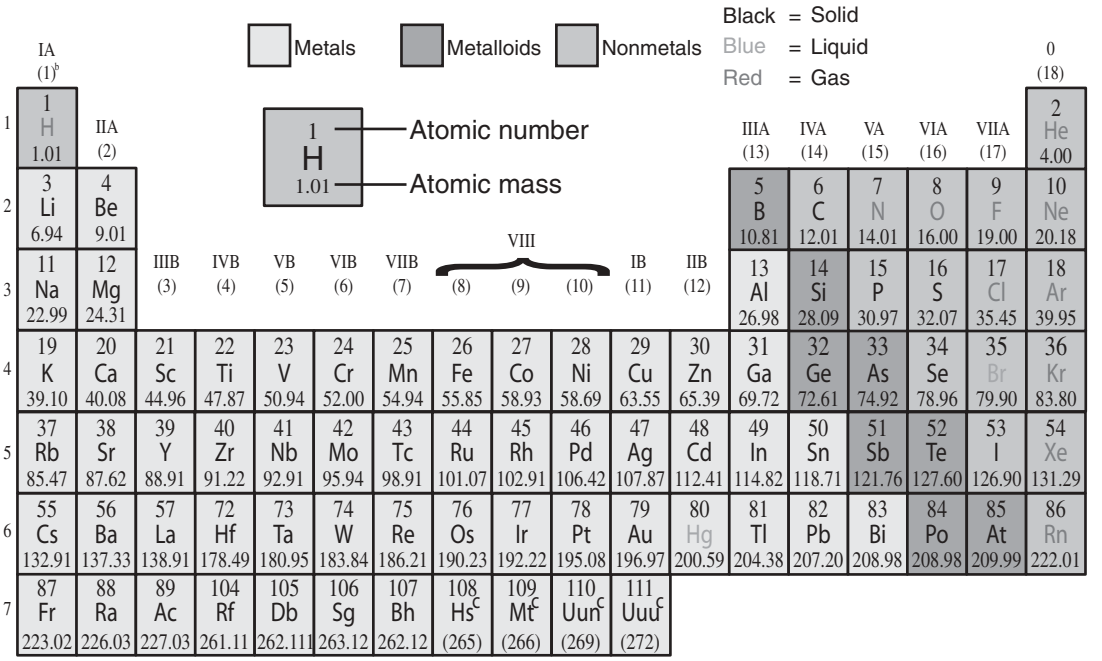
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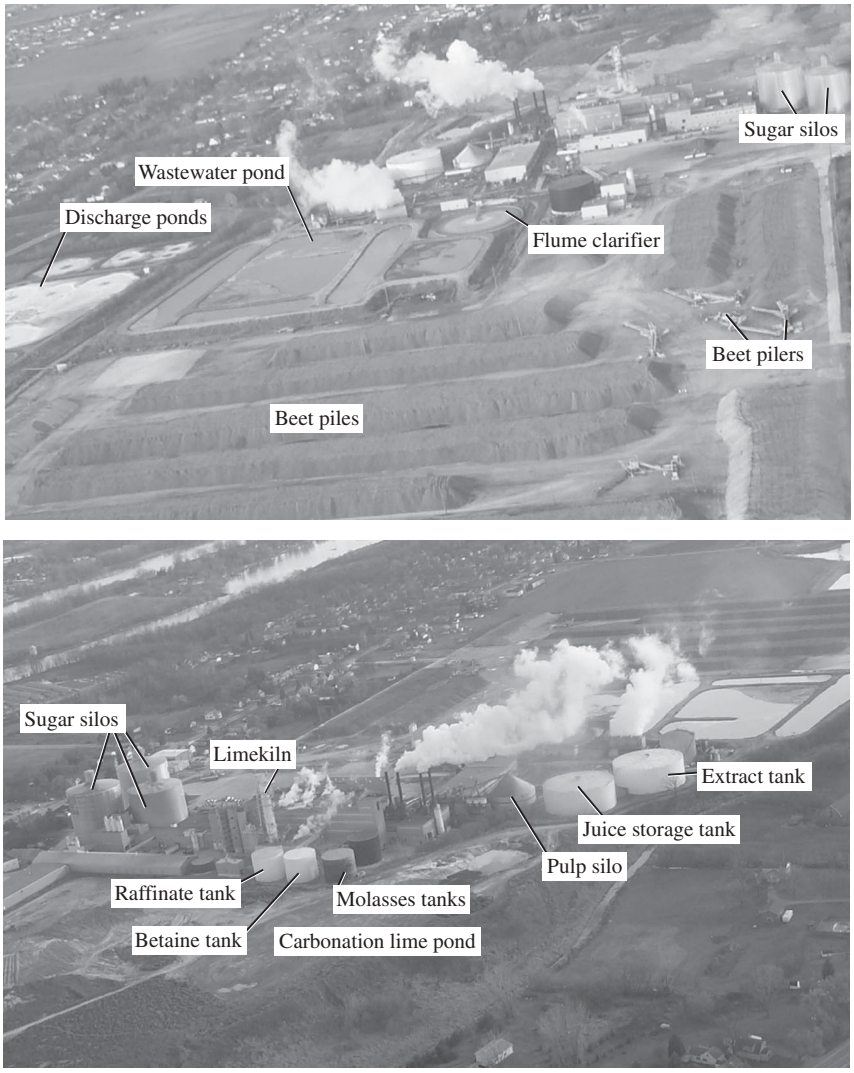
Periodic table of the elements



58 Ce 140.12	59 Pr 140.91	60 Nd 144.24	61 Pm 144.24	62 Sm 150.36	63 Eu 151.97	64 Gd 157.25	65 Tb 158.93	66 Dy 162.50	67 Ho 164.93	68 Er 167.26	69 Tm 168.93	70 Yb 173.04	71 Lu 174.97			
90 Th 232.04	91 Pa 231.04	92 U 238.03	93 Np 237.05	94 Pu 244.06	95 Am 243.06	96 Cm 247.07	97 Bk 247.07	98 Cf 251.08	99 Es 252.08	100 Fm 257.10	101 Md 258.10	102 No 259.10	103 Lr 262.11			

Elements mostly used in sugar technology

Element	Symbol	Atomic mass
Aluminum	Al	26.98
Calcium	Ca	40.08
Carbon	C	12.01
Chlorine	Cl	35.45
Copper	Cu	63.55
Fluorine	F	19.00
Helium	He	4.00
Hydrogen	H	1.01
Iron	Fe	55.85
Magnesium	Mg	24.31
Mercury	Hg	200.59
Nitrogen	N	14.01
Oxygen	O	16.00
Potassium	K	39.10
Silicon	Si	28.09
Sodium	Na	22.99
Sulfur	S	32.07



A beet-sugar factory from two sides
(Courtesy, Michigan Sugar Company)